Gas Journal

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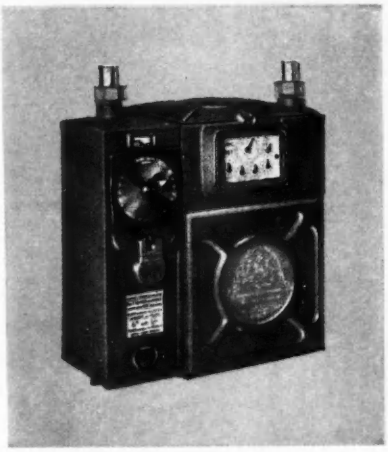

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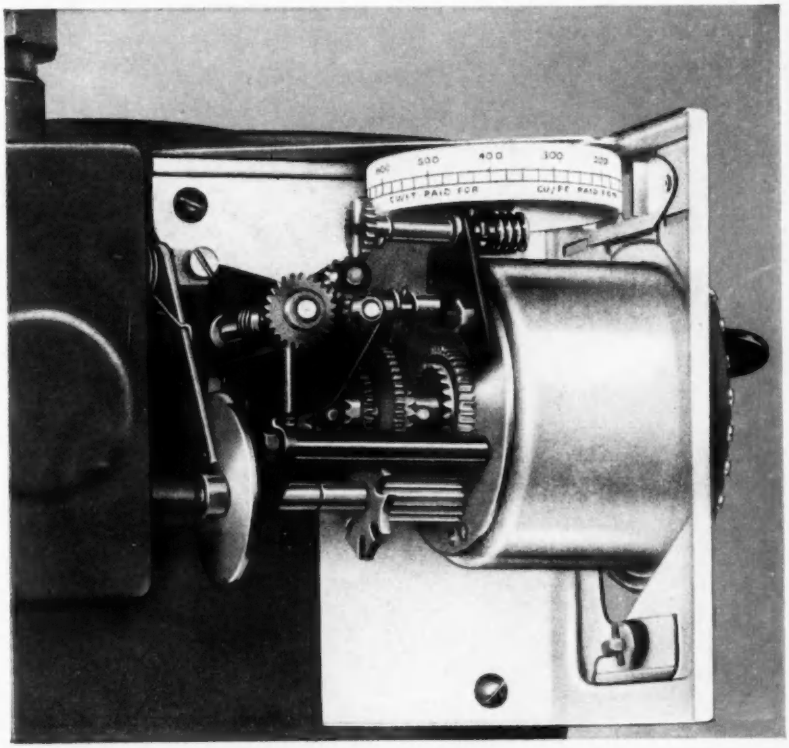

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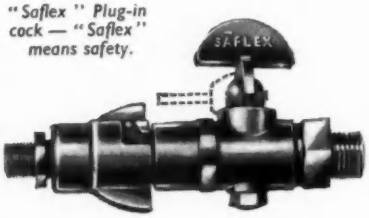

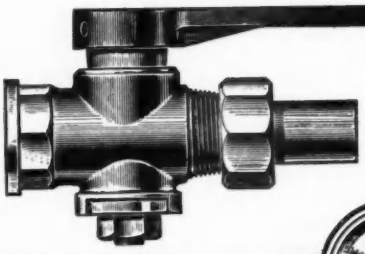

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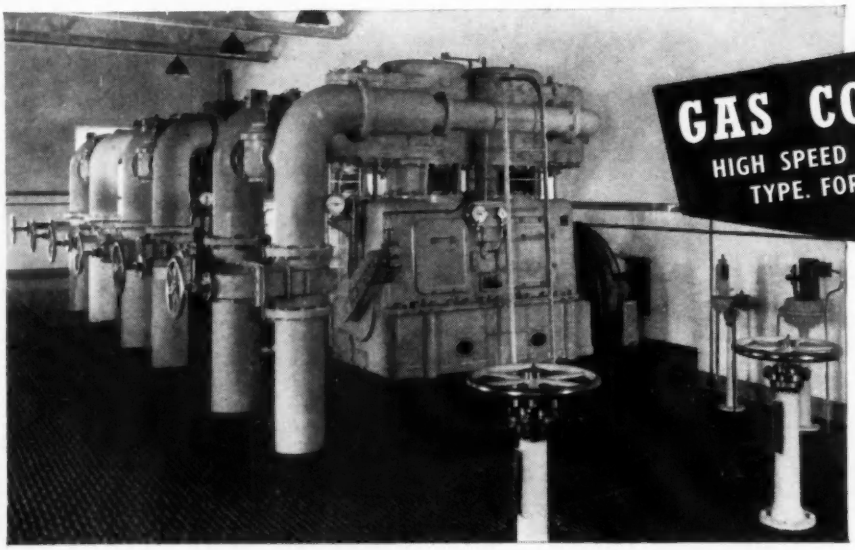

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Gas Journal

VOL. 297 NO. 4984 JANUARY 21, 1959

111TH YEAR

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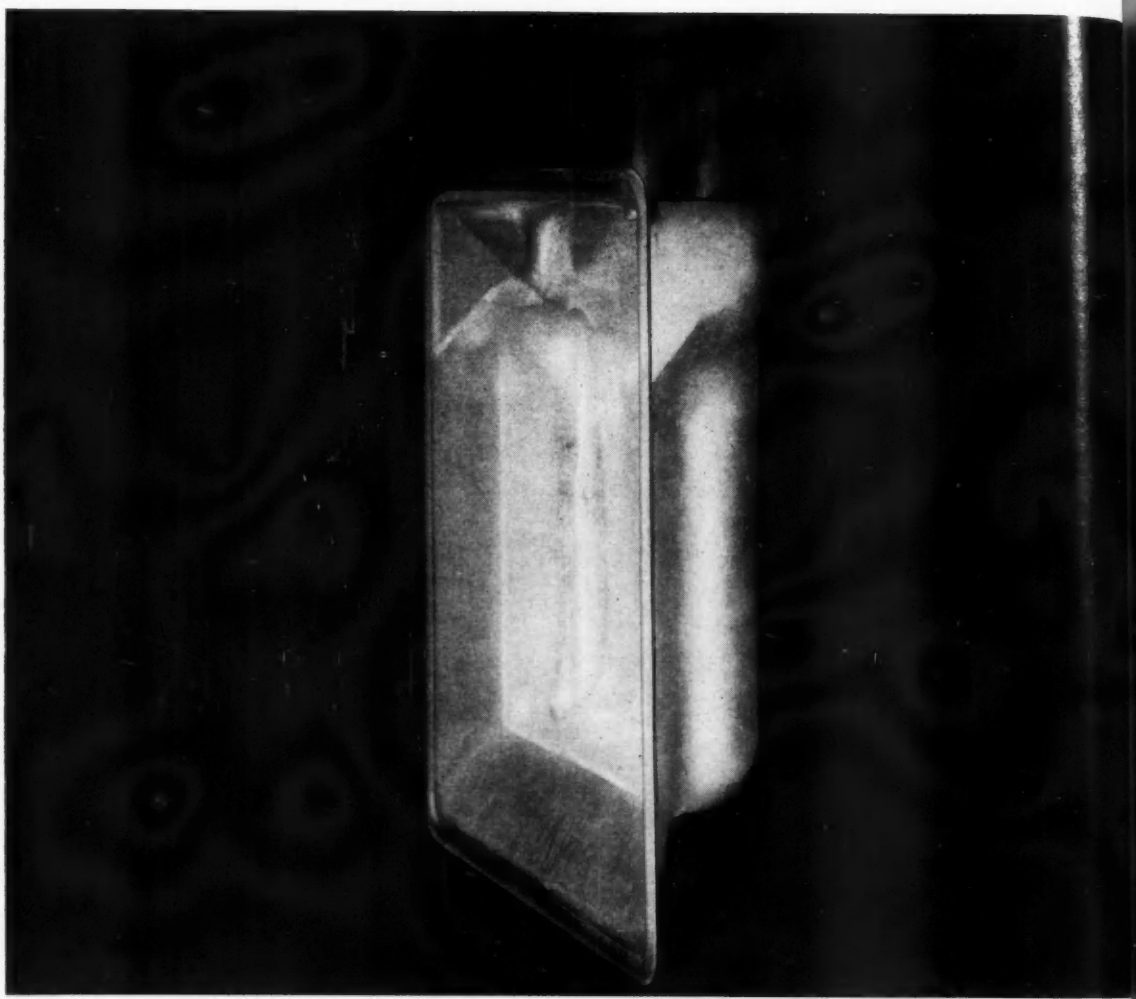

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Gas Journal

VOLUME 297. No. 4984 JANUARY 21, 1959. 111th YEAR

Facing Up to Effluent Disposal

THE bane of chemical processing industries is the disposal of effluents or trade wastes. The gas industry also has its effluents to dispose of and these consist of ammonia liquor in various forms.

Although the problems of effluent disposal must have faced the industry from its earliest days, surprisingly little is heard of what steps were taken to solve them. Presumably local streams, rivers or canals were used to carry away unwanted liquids, since the well-organised, central sewage systems of today would not have been available or indeed in existence in those early days.

The demand for sulphate of ammonia as a fertiliser before the first world war made possible an economic outlet for the ammonia being produced at gasworks and coke oven plants. But with the rise of the synthetic ammonia industry by the exploitation of the Haber process in this country after the 1914-18 war, the price obtainable for ' by-product ' ammonia fell and sulphate manufacture became largely uneconomic. It was only possible to continue its production in the larger coke oven works and the very largest gasworks, making full use of the selling agency that had been set up for its disposal to farmers and others throughout Great Britain and Ireland.

Many of the smaller gasworks were able to dispose of their ammonia liquor to the local authorities' sewers by special arrangement and payment of a fee which varied with the locality and the volume to be disposed of. With nationalisation and the concentration of gas production units, the disposal of ammonia liquor became increasingly difficult since the volume to be dealt with at the sewage out-fall stations threatened to exceed the spare capacity that the local authorities had made available. The increase in housing after the second world war and the ever-rising standards of living aggravated the sewage disposal problem even further.

When sulphate of ammonia manufacture proved uneconomic, the production of concentrated ammonia took its place at some works, but the spent ammonia liquor from such plants had still to be disposed of. Although the spent liquor from the coke oven sulphate plants may have been stripped of both ' free ' and ' fixed ' ammonia, that from the gasworks' concentrating plants still contained the ' fixed ' ammonia. Added to this, there were also present in this liquor certain toxic constituents, such as phenols, cyanides and thiocyanates, which caused a high oxygen absorption factor. Since this may be taken as a measure of the effect of the liquor on the sewage treatment plant, it was necessary to keep to a minimum and within very careful limits. With new legislation passed after the second world war, preventing the pollution of natural water courses and canals by trade wastes and the strengthening of the powers of river boards, the gas industry was prevented from the disposal even of these spent liquor effluents in this way.

What could the industry do to overcome these very pressing disposal problems? A great deal of research had been going on in both the carbonising industries to find ways of rendering these spent liquors innocuous. There are three outstanding examples of where the problem was tackled on a large scale and where suitable treatment plants have been built. There are at Tingley and Denton gasworks and at the Avenue coking plant of the National Coal Board. The methods employed differ in each case, but the resultant effluent is perfectly satisfactory and can be accepted by the local authority's sewage system. The oxygen absorption factor of the effluent has been reduced to such a low figure that it will have no harmful effects on the treatment of the normal domestic sewage. Such treatment of spent liquor is expensive and it is generally accepted that if a small profit could be made on the production of concentrated ammonia, it would be lost on treatment of the effluent.

So costly are the processes, in fact, that the North Eastern Gas Board has been compelled to close down the Tingley plant. Where a gasworks is near a seaboard, the ammonia liquor may be directly disposed of into the sea, where it is quickly diluted and does no harm. Care must be taken to discharge it on the ebb tide and this is being most ingeniously done with the aid of tide clocks from the Lostock Hall works at Preston. The proximity of the sea will greatly assist the disposal of liquor from the Lurgi plant now being built at Westfield in Fife. The volume of liquor from such a plant is likely to be large, so this particular means of disposal was fortunate.

Much work has been done on the direct spraying of agricultural land with ammonia liquor. Here again the economics of the process must be carefully considered, since transport costs by road tanker are heavy, and it has been definitely established that 20 to 30 miles from the gasworks is the maximum radius over which liquor spraying is economic. Under no circumstances is it profitable to work from gasworks in built-up areas.

It is quite clear that such problems must be solved by the industry and it is equally clear that it is using every means to accomplish this end. Research continues at the Gas Council Research Stations and the University of Leeds. Much work has been done on the pretreatment of liquor before it leaves the gasworks or coke oven plant, but before such treatment can become effective, a complete knowledge of the liquor's constituents and a reliable analysis are needed. In a paper we publish this week by Dr. L. Barker, and Mr. N. W. Hollingworth, both of the University of Leeds, on 'an Investigation into the Composition of Ammonia Liquor,' the most up to date analytical methods, including chromotography are applied. We think that the inclusion of tables showing the analysis of the most commonly met with liquors from all three types of retort installation, low temperature plants, coke ovens and a Lurgi plant in Germany, are of the greatest interest. The very complete analysis of mono- and polyhydric phenols must be unique. Such information should be of great assistance to gas chemists and gas engineers who may thus be able to modify the working of their plants or may consider keeping liquors from certain sections separate to facilitate their treatment. The paper, which is the second in a series by these authors, may appear a little academic but this, we are certain, should not deter gas engineers from applying the results.

Growing Pains

A SITUATION has arisen in the transport of oil and natural gas across Canada that at once brings home to the British reader the size of the North American Continent from East to West and the sparseness of the population in the northern half of it. We have heard much talk lately about discoveries of oil and natural gas in the Prairie Provinces of Alberta and Saskatchewan, and parts of Northern British Columbia. Pipe-lines have been constructed to transport both gas and oil eastward from Alberta. Running east are the Trans-Canada natural gas pipe-line to Montreal and the Interprovincial oil pipe-line to Toronto. Running westward is the Trans-Mountain pipe-line carrying oil through the Rockies, with a natural gas pipe-line, the West Coast line, running from Northern British Columbia southward.

Naturally the oil producers in Alberta are anxious to improve their competitive power in the Montreal market where crude is refined and from which products are distributed, and a small group of producers proposed building a new pipe eastwards to Montreal for this very purpose. The idea was opposed by the Montreal refiners on the grounds that Canadian crude is not competitive with sea-borne crude from Venezuela and the Middle East. Suggestions that import quotas, an import duty or compulsory legislation for the use of domestic crude oil, should be imposed to restrict the import of foreign crudes, met with no more sympathy from 95% of the Alberta producers.

The Borden Commission, which is investigating the whole question of the export of natural resources requested an independent survey to find out whether an extension of the Interprovincial pipe-line from Toronto to Montreal or a duplicate pipe-line, would deliver oil the more cheaply. At an average daily delivery of 250,000 barrels, the extension would transport the oil at a saving of nearly 5 cents per barrel and at lower deliveries the saving would be greater. The new pipeline would only be economic at much greater oil flows. This at once suggests that the available pipe-lines are working at too low a load factor, with the heavy capital charges and other standing expenses not being adequately earned by oil deliveries at a competitive price. This is borne out by the Trans-Mountain, a 24-in. pipeline, considered at the time as the smallest economical size which could be laid through the difficult mountain country westward from Alberta. It took 3½ years after completion during the Suez crisis before the line was used to full capacity. It is unlikely to be so again in the foreseeable future. The Trans-Canada natural gas pipe-line running between Edmonton and Montreal is now carrying about 100 mill. cu.ft. of gas a day but has a capacity of a further 200 mill. cu.ft. per day, should the need arise. The West Coast pipe-line is also working below capacity, carrying natural gas from Northern British Columbia.

What is the solution to Canada's problem? Fuel reserves are in not easily accessible places and there are few potential consumers. The solution seems to be to work up an export market for both oil and natural gas to the Northern United States. The permission to do this lies with the Canadian Government on the recommendation of the Borden Commission which will decide in due course whether indigenous mineral reserves should be exported or not.

The United States would welcome any such move since the cost of importing oil from Canada on its border would be considerably cheaper than bringing oil from the South. British Columbian natural gas could implement supplies now being brought by the Pacific North West pipe-line from California and the Deep South. The interested parties in the U.S.A. have not in fact been slow to criticise the Canadian Government's delay in making up its mind. On the other hand, we think that a fast-developing country, rich in mineral wealth, should be careful to conserve its fossil fuels to assist in its expansion and to meet from its own resources the greater demands which are likely to be caused by an increasing population, with rising standards of living.

Personal Notes

MR. ARNOLD CARR, Deputy Chairman of Thos. W. Ward, Ltd., Albion Works, Sheffield, has been appointed Chairman of three Ward subsidiaries, Thomas Smith & Sons (Rodley), Ltd., excavator and mobile crane manufacturers, John Smith (Keighley), Ltd., makers of overhead travelling cranes, and Widnes Foundry & Engineering Co., Ltd., makers of iron castings, fabrications, etc. This appointment is consequent upon the death of MR. FRANK R. STAGG, who was for many years chairman of these three companies.

MR. R. A. BENT has been appointed to the Board of Lancashire Dynamo Holdings, Ltd. Mr. Bent, who was born in Stafford in 1922, is Managing Director of Lancashire Dynamo Electronic Products, Ltd., Rugeley, a Company of which he was one of the founders, in 1946.

MR. W. R. DRURY has been appointed the West Midlands, Northern and North Wales areas representative of F. A. Borchardt Ltd., for the distribution of DRU-gasar balanced flue heaters, the Arnhem closed heating stove, etc. The appointment is in addition to Mr. Drury's representation in the North Western area and he will continue to operate from 142, Oxford Road, Manchester, 13.

MR. E. K. STEWART-SMITH, M.B.E., of Halstead, Essex, has been re-appointed part-time member of the Eastern Gas Board.

MR. G. A. WOOD, Station Engineer, Paisley district, Scottish Gas Board, gave a talk to fellow-members of the Paisley Rotary Club recently on the gas industry.

Mr. W. R. Branson, Deputy Chairman of the West Midlands Gas Board recently attended a 'parents evening' at Dartmouth Street School, Birmingham, where many of the Birmingham Division's future craftsmen take their training. The evening was organised by Mr. W. J. Parsons, Birmingham Division Sales and Service Officer. Pictured above: Graham V. Cooper receiving from Mr. Branson a special Board prize for being judged the best pupil in the first year of his course.

MR. J. W. LEE has been appointed Sales Manager of Fluidrive Engineering Co., Ltd. The appointment is in connection with the reorganisation and expansion of the engineering sales department, under MR. W. H. K. JAMES. MR. A. W. R. WILLIS becomes Chief Application Engineer (Motors) and MR. R. V. ADAMS, Chief Application Engineer (Engines).

MR. A. H. CATTON has been appointed to part-time membership of the North Eastern Gas Board. He takes up his appointment on May 1 this year. Mr. Catton is Managing Director of Catton & Co., Ltd., Leeds.

Obituary

MR. JOHN DEMAIN, of Pinchbeck, Lincs., who was for nearly 20 years gas and water engineer of Spalding (Lincs.) Urban District Council, retiring in March, 1946, has died at the age of 77. Prior to moving to Spalding in 1928 he was gas and water engineer and manager at Ripon, Yorks.

SIR CLAUDE D. GIBB, who was to have been awarded the 1959 James Watt International Medal by the Council of the Institute of Mechanical Engineers, has died. The award was to have been made at the meeting of the Institution on January 23. At the meeting an appreciation of the work of the late Sir Claude will be given, and arrangements announced for the transmission of the medal to Lady Gibb. Sir Claude was nominated by the Institute of Engineers, Australia, and the Institute of Mechanical Engineers.

MR. CHARLES GEORGE GRIMWOOD, former managing director of Sudbury gasworks, died recently at the age of 85. He retired from his managing directorship shortly after nationalisation in 1949, having given 60 years service to the industry.

MR. C. DUNCAN, Service Engineer and Manager at Huntingdon for the Eastern Gas Board for the past 12 years, has retired, for health reasons, at the age of 63. The Board recently began providing gas for the Huntingdon area from the Cambridge grid main and accordingly it is not proposed to appoint any successor to Mr. Duncan.

Apprentices prize distribution day

ROBERT DEMPSTER & SONS, LTD., Elland, held their annual apprentices prize distribution in the firm's canteen recently, which was attended by a large gathering of apprentices, parents and friends.

Guests included the Venerable Archdeacon Eric Treacy, M.B.E., Vicar of Halifax, Mr. H. Schofield, Headmaster of the new Technical High School at Bradshaw, Halifax, and Mr. Horace Whiteley, President of the Halifax and District Chamber of Commerce.

Mr. B. C. Morton, Managing Director of Robert Dempster & Sons, Ltd., extended his welcome to those attending, congratulated the successful apprentices, and offered a word of encouragement to those who had not been so fortunate. Speaking of education he expressed his concern regarding the lack of provision for craft technical training in urban comprehensive schools.

Archdeacon Treacy presented prizes to the following successful apprentices:

Gas Plant Manufacturers Development Council prizes: P. Dean, G. Littlewood, R. A. Childs, D. R. Holden, and S. G. Hagreen.

Mechanical Handling Engineers' Association Prizes: D. Sykes, M. Taylor, and M. Mellor.

Robert Dempster & Sons, Ltd., prizes: R. Lewis, D. Haigh, R. Walker, B. Lancaster, and J. Nelson.

Certificates of apprenticeship were presented to D. Haigh, V. Chapman, and R. Waddington.

Diary of Forthcoming Events

January 22.—INSTITUTE OF FUEL (EAST MIDLAND SECTION): Gas Showrooms Lecture Theatre, Nottingham. 'Smoke Abatement—The Clean Air Act and the Carbonisation Industries,' by C. E. Hall. Meeting held in conjunction with the National Society for Clean Air, East Midlands Division. 6.15 p.m.

January 24. — WESTERN JUNIORS: Taunton. Talk by Mr. A. R. Bovington on his experiences in the U.S.A. as a recipient of the Woodall-Duckham Educational Award of the I.G.E. 2.15 p.m.

January 27.—WALES AND MON. JUNIORS: Aberdare. 'The Construction of Small Diameter High Pressure Grid Gas Mains,' by H. Breeze and S. H. P. Nelson.

January 28.—SOCIETY FOR ANALYTICAL CHEMISTRY, LONDON MICROCHEMISTRY GROUP: 'The Feathers,' Tudor Street, Fleet Street, London, E.C.4. Discussion on 'The Determination of Small Quantities of Toxic Substances in the Atmosphere.' 6.30 p.m.

January 29.—EASTERN JUNIORS: Spalding. 'Modern Methods of Leakage Detection in Mains and Services,' by E. R. Fitch.

February 3.—SOUTH EASTERN G.C.C.: Caxton Hall, Westminster.

Ammoniacal Liquor Fertilisers, Ltd., one of whose tankers was illustrated on p. 611 of our December 31 issue, inform us that nowhere in the area of the North Eastern Gas Board is gas liquor diluted as was implied.

GAS POSTER PAINTING COMPETITION PRIZES

Winning entry was of 'professional standard'

THE creative director of a leading London advertising agency said recently that the winning entry in a gas poster painting competition was 'right up to professional standards—in fact, better than some.' He was Mr. Leslie Porter, of the London Press Exchange, who was one of the judges of the competition which was organised for children by the Hulton Press junior publications and the Gas Council. He was speaking at Hulton House, Fleet Street, London, where Sir Harold Smith, Chairman of the Gas Council, presented prizes to the major award winners.

More than 5,000 posters were entered in the competition, which was divided into three age groups: 14-16, 11-13 and 6-10. First, second and third prizes of £25, £15 and £10 respectively were awarded in each of the three groups, and in addition there were 350 £1 consolation prizes. Among the entries were 150 from 22 schools which made block entries.

The exhibition hall at Hulton House on Thursday was bright with the colours of all 359 award-winning posters, when the major prizes were presented. The exhibition will remain open to the public for a week.

A 16-year-old apprentice printer, who attends night classes in order to study art, achieved the distinction of being first in the 14-16 age group, which meant that his was considered to be the best poster in the competition. He was Trevor Varley, whose home is at Leeds.

Mr. Porter commented that he would like to see some of the poster ideas used by the winners adopted.

Before presenting the awards, Sir Harold Smith said the gas industry felt that it was doing a really worthwhile educational job in telling young people how gas appliances work and what they can do to make life easier and more comfortable. 'The Poster Painting Competition is a part of our campaign and if the prizewinners know as much about gas as they know about designing and painting posters, there isn't a great deal we can teach them,' he said.

Left: *Trevor Varley (16), who gained the top award in the Hulton Press-Mr. Therm Poster Painting Competition, chats with Sir Harold Smith, Chairman of the Gas Council, during the presentation of prizes. Trevor's poster—with his own slogan 'You too can rely on gas'—was first in the 14-16 age group of awards.*
Above: *The poster entered by Diane Proctor, (10) which gained for her the first prize in the 6-10 age group.*

Leeds may have largest smoke control area

LEEDS Corporation Health Committee is expected to instruct the making of an Order establishing the biggest smoke control area in the country.

It will take in about 10,700 buildings, about 5,000 of which are houses, many of them requiring new appliances for burning smokeless fuels. The Order will require the approval of the City Council as well as formal confirmation from the Ministry of Housing and Local Government.

Next year

In view of the size of the proposed smoke control area it is intended to wait until July or August next year before the Order takes effect.

BREAK-IN DAMAGE—BOARD MUST PAY

A KIRKCALDY (FIFE) man has successfully fought the Scottish Gas Board over their claim that he should pay £1 8s. 11d. for repairs to a meter damaged by a burglar.

Appearing at Kirkcaldy Sheriff Court for the Board, Mr. W. S. Duncan, said that his clients were prepared to go to proof because it was a matter of principle to them. They declared that in a list of tariffs drawn up and published nationally in July, 1957, they were empowered to hold consumers responsible for damage, loss or theft of meters and fittings supplied by them, however such loss or damage might be caused.

But Sheriff J. Sinclair Shaw found in favour of Mr. William Shields, 144b, St. Clair Street, and awarded him expenses of the action.

Mr. Shields told the court that his house had been broken into and the gas meter forced in September, 1957, while he was out with his wife.

'Abrogation'

His solicitor, Mr. A. T. Wood, submitted that, though there was nothing in the Gas Act making consumers responsible for damage to appliances held by them, the Board had attempted to abrogate its own responsibilities by publishing a tariff in newspapers. In this tariff they passed liability on to the consumer.

Responsible

In addition, they had brought out a form relating to applications for a supply of gas. New consumers were given this form to fill in so that they could be held responsible for damage or theft of meters.

CANVEY ISLAND

Insulation of pipe-lines to carry methane must be thorough to prevent boiling-off

THE experiment being conducted on Canvey Island on the technical and economic feasibility of importing methane from the transatlantic oilfields involves piping the liquid into two insulated storage tanks, from which it will be drawn as required, evaporated and fed into the normal town gas system.

VAPOUR LOCKS

One of the many problems to be surmounted is to prevent the methane from boiling off, which it will do at normal pressure if its temperature rises to −258°F. In the pipe-line carrying the methane from ship to storage tank, very thorough insulation is necessary, to prevent undue evaporation and the formation of vapour locks. The 10-in. aluminium alloy main, about 2,000 ft. long, is covered with 6 in. of Onazote cellular insulation, which has a thermal conductivity of almost half that of cork. To ensure that this performance is maintained, it is essential to keep the insulation absolutely dry for, as with all thermal insulation systems, full thermal efficiency depends on a complete and lasting water- and moisture-proof seal. Although Onazote itself has a very low moisture vapour transmission, moisture must be prevented from entering the joints between adjacent sections. This installation is particularly vulnerable because of the combination of moisture-laden sea air and the extremely low temperature of the pipe-line which would encourage condensation. The measures taken to ensure long-term moisture-proofing of the exposed line are interesting.

Onazote, manufactured by the Expanded Rubber Co., Ltd., of Croydon, and applied by the Onazote Insulation Co., Ltd., is fitted to the pipe in accurately shaped longitudinal sections (Fig. 1), each horizontal and vertical joint being sealed with a strip of 2-in. wide Denso tape. This material, made by Winn & Coales Ltd., Chapel Road, London, S.E.27, consists of an absorbent cotton fabric fully impregnated and coated with a stable compound based on saturated petroleum hydrocarbons. The compound incorporates inert siliceous fillers to provide the required body, and special corrosion inhibitors, while bactericides prevent attack on the fabric itself.

BY HAND

The compound is tacky, and adheres to any dry material. After the joints have been covered, the whole surface is liberally smeared with Denso paste, which is similar to the compound used for impregnating the tapes. The jelly-like paste is easily applied by hand, and seals all the pores and fissures in the insulant. It also provides a base for the final covering, which consists of a 'bandage' of Denso tape, spirally-applied in widths varying from 2 to 12 in. depending on the contours of the pipe. The compound applied as paste and that in the tape merge into a homogeneous thick skin, which is completely impervious to moisture and chemical attack.

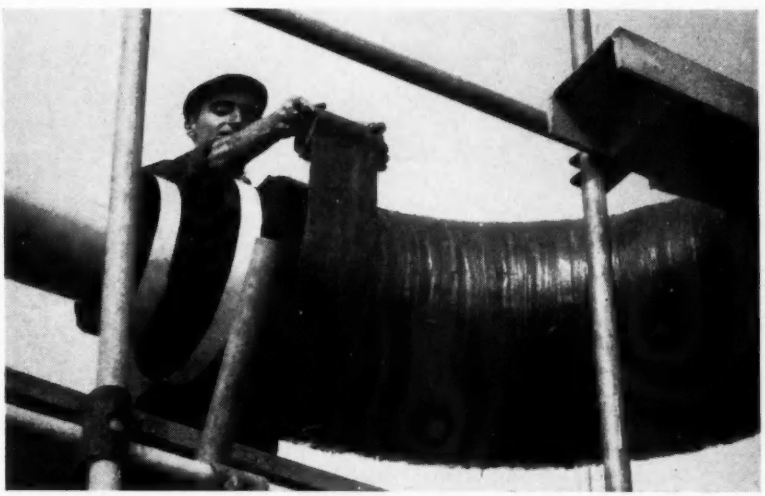

Fig. 1. All joints in the three 2-in. layers of Onazote insulation of the methane main are sealed with Denso tape, the whole installation then being sealed with a layer of Denso paste and a further continuous bandage of tape, as shown here.

ALUMINIUM PAINT

Mechanical protection is given to the finished insulation by a final covering of bituminous felt, held in place with wire mesh and painted with aluminium paint to reduce absorption of the sun's rays.

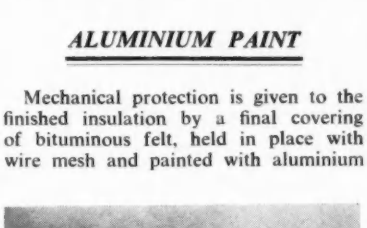

Fig. 2. The tape is used to provide temporary protection on unfinished work. At the temperature of −258°F., the presence of any moisture would result in prohibitive loss of insulation efficiency. Being non-setting, the tape is easily removed when required.

One of the most useful properties of the Denso materials is that they retain their tackiness indefinitely, and do not harden. They can thus be removed, even after some years' service, without damage to the underlying insulating material. They are thus far superior to the traditional hot bitumen applied as a protective, which must be chipped off and the cork or other material broken away, should it be necessary to carry out maintenance on the actual pipe work.

Advantage was taken of this non-setting property when temporary protection was required. In Fig. 2, a section of the pipe-line has been left uninsulated while awaiting the fitting of a flange. The insulation has been tapered off in layers so that, when completed, there will be no continuous radial seam.

NO MOISTURE

To make sure that no moisture penetrates the joints before the flange is fitted, the section has been bandaged with Denso tape which will simply be stripped off when the flange is ready to be fitted. (The flanges are subjected to the most stringent cold tests before installation, careful observations being made during the tests.) All exposed ends are treated in this way when work is finished for the night. The tape removed to permit fitting the flanges can usually be reused for the final job, thus preventing wastage.

From a paper to the Yorkshire Junior Gas Association, Sheffield, November 22, 1958.

Town Gas Desulphurisation by Oil Washing

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and

P. G. HARRIS,

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THE removal of organic sulphur from coal gas is a problem which confronts those concerned with gas production. While total elimination is desirable, it will be appreciated that economics enter into the problem. The policy of the East Midlands Gas Board is, therefore, to reduce the organic sulphur compounds to the minimum. This paper is an attempt to set down actual working results obtained on our particular plant, using routine testing methods and normal operational technique. The tests were carried out under widely varying conditions in order to show the flexibility of the plant and to examine the results obtained under these conditions. While testing is not yet complete, we thought that the results so far obtained were sufficient to give a fair knowledge of plant behaviour and, we hope, will be of some benefit to either those who operate this type of plant and have not yet had time to investigate plant conditions, or, to people who may have this type of plant under their control in the future.

The desulphurising plant, built by W. C. Holmes & Co., Ltd., for the East Midlands Gas Board, Car House works, was first commissioned in September, 1956. The plant, which is in two complete units, is designed for 95% benzole removal and 90% extraction of the organic sulphur compounds which are removable by oil washing, from 13.5 mill. cu.ft. per day of coal gas from intermittent vertical chambers, 3 to 5 mill. cu.ft. per day of crude coke oven gas and up to 4 mill. cu. ft. per day of carburetted water gas. The position of the naphthalene and desulphurising scrubbers in relation to the ancillary plant can be seen in Fig. 1. The mixed gas stream passes through the naphthalene removal tower, 9 ft. diameter by 50 ft. high. This tower, which is constructed of mild steel plates riveted together, is filled with timber laths, closely spaced to give even distribution of gas to ensure maximum wetted surface area. Above the timber lath filling is a Silver type distributor for even distribution of the oil throughout the tower. Following the naphthalene scrubber, the gas is split into two separate streams and treated for removal of H_2S by iron oxide in tower purifiers. The two streams of purified gas are treated for benzole and organic sulphur removal before passing to the holders. The two tower scrubbers are 95 ft. high and are of similar design and construction to the naphthalene removal tower.

The naphthalene scrubber is designed to remove over 90% of the naphthalene in the gas, with a throughput of 22.5 mill. cu.ft. per day and, for 80% removal at peak loads, of 25.5 mill. cu.ft. per day. In actual practice, the naphthalene removal, with a gas throughput of 19 mill. cu.ft. per day, is 95% and, with maximum throughput of 25 mill. cu.ft. per day, the efficiency of extraction is 88%. The oil, recirculated in the tower, is stored in a mild steel cylindrical tank 10 ft. diameter by 10 ft.

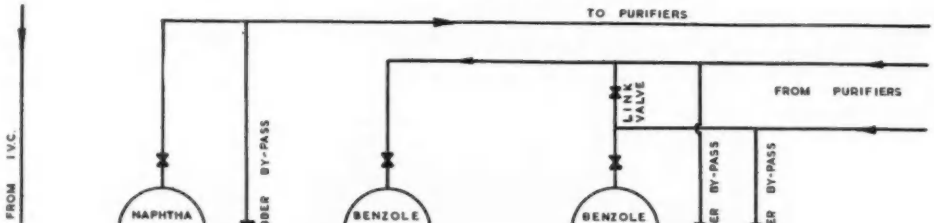
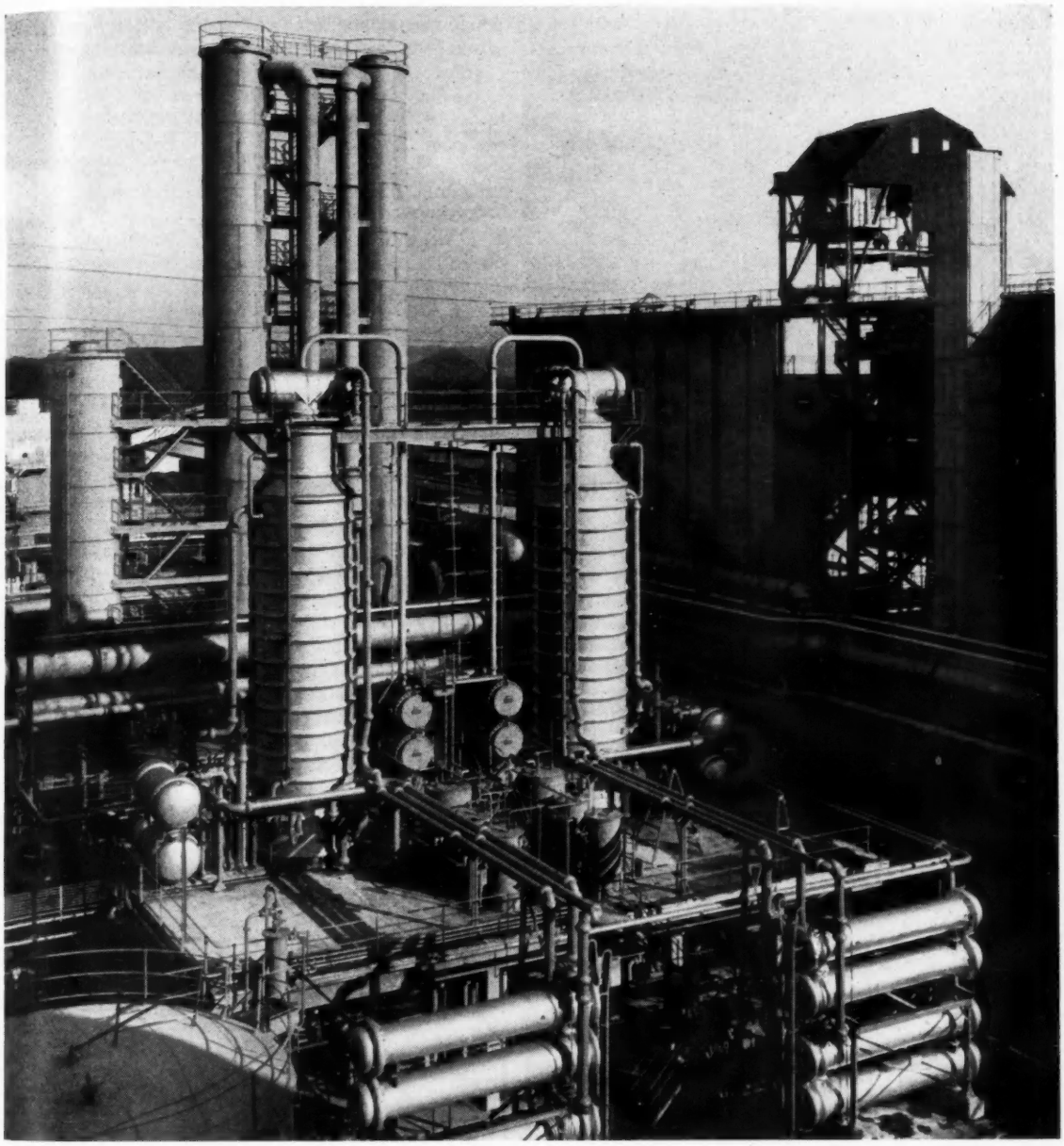


Fig. 1 Diagrammatic arrangement of gas mains and tower scrubbers.



A general view of the benzole recovery and desulphurising plant, with the benzole scrubbers in the background. To the right is the tower box purifier plant. (By courtesy of W. C. Holmes & Co., Ltd. Woodall-Duckham Construction Co., Ltd., were the main contractors.)

long, fitted with a float box controlling the flow of make up oil from the desulphurising plant to maintain a constant volume of oil in circulation. Two centrifugal pumps, one normally acting as a standby, recirculate the oil at a rate of 7,500 gal. per hour. A rich oil bleed from the naphthalene tower circuit of 300 gal. per hour into the benzolised oil tanks, is needed to provide maximum removal efficiency under peak load conditions.

In the event of benzole removal from the gas being impractical, a procedure which is discussed later on in the paper, the rich oil bleed can then be (1) regenerated continuously in one of the benzole stripping stills, or (2) regenerated intermittently, as required by the saturation of the oil in circulation, or (3) disposed of in the carburetted water gas plant and replacing it by an equal volume of fresh oil. The last procedure is preferred due to the uneconomical running of the stripping still for oil regeneration only.

Vacuum desulphurising plant. Fig. 2 shows the arrangement of one of the units and, for ease of description and correlation of operational data, the remainder of this paper will deal specifically with one plant only.

The naphthalene and desulphurising scrubbers are operated on the counter flow system for maximum efficiency of removal. Spray traps are provided on the outlet main from the desulphurising towers and the oil collected is bled off through a seal to the towers. The benzolised oil flows by gravity from the bottom of the

desulphurising tower and is stored in a mild steel cylindrical tank of 8,000 gal. capacity. The tank has provision for draining off any water which settles out from the oil and also provides a gravity feed to the benzolised oil pump, which circulates the oil through the various heat exchangers to the stripping still. The pump is capable of delivering 15,000 gal. of oil per hour.

Hot Reflux Condenser

Heat exchangers. The initial preheat imparted to the benzolised oil is achieved by the vapour to oil heat exchanger which is mounted on the top of the stripping still and is in the form of a hot reflux condenser, which is of shell and tube design and, under normal working conditions, imparts 20°C. of preheat to the benzolised oil. Incorporated with the heat exchanger and positioned beneath it, is a separator which delivers the condensed naphtha saturated oil leaving the exchanger, either to the stripping still beneath, or to the naphtha/water separator. The next preheat stage is the oil to oil heat exchangers, also of the shell and tube design, and installed to enable a portion of the heat in the debenzolised oil leaving the still to be transferred to the benzolised oil, leaving the vapour to oil heat exchangers. The benzolised oil flows through the tubes, around which flows the debenzolised oil. The third and final preheat stage is in the form of an indirect steam heater, again of the shell and tube design. The benzolised oil flowing through the tubes, around which steam is admitted. In order that a constant oil temperature of 80°C. is maintained at the heater outlet, an automatic steam valve is fitted on the steam inlet which is operated by means of the oil temperature. Suitably preheated, the hot benzolised oil enters the top of the stripping still where live steam, admitted at the bottom, extracts the crude benzole from the oil as it gravitates down the still.

TABLE 1

Normal temperature conditions throughout the oil system

Plant position		Benzolised oil temperature °C	Debenzolised oil temperature °C
Out scrubber		26°	
Vapour/oil heat exchanger	In	27°	
	Out	46°	
Oil/oil heat exchanger	In	46°	76°
	Out	49°	72°
Steam heater	In	49°	
	Out	80°	
Rack oil cooler			72°
			28°
Inlet scrubber			28°

Gas Temperature 26°C

Stripping still. The vacuum stripping still is constructed of cast iron, is 9 ft. diameter and contains 12 trays, 16 in. deep, each tray being fitted with a series of bubble hoods. It can be seen from the flow diagram, Fig. 2, that two float valves are fitted on the still, controlling the flow from the benzolised and debenzolised oil pumps respectively, thus ensuring that a balance is maintained between the oil entering and leaving the still. The still is designed to operate under 10 in. mercury absolute pressure conditions, the effect of which will be seen later. The hot debenzolised oil, on leaving the base of the still, gravitates to the debenzolised oil pump which circulates the hot oil through the oil to oil heat exchangers and rack coolers to the oil inlet at the top of the desulphurising scrubber. The pump is a five stage centrifugal pump and, like the benzolised oil pump, is capable of circulating 15,000 gal. oil per hour, being

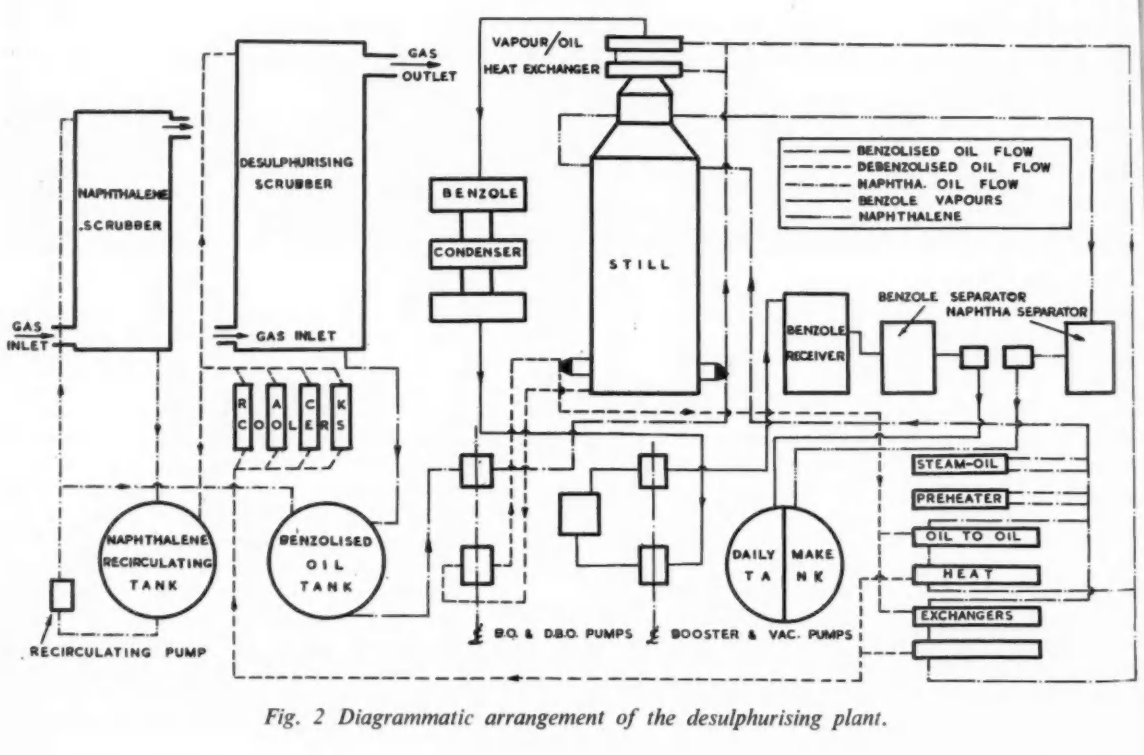


Fig. 2 Diagrammatic arrangement of the desulphurising plant.

mounted on an extension of the corresponding benzolised oil pump bedplate and driven by an electric motor through a common shaft. Since the pump is dealing with oil around 76°C. the bearings are water cooled. The suction glands of both the benzolised and debenzolised oil pumps are sealed with oil, fed from an external tank. The pressure gland of the benzolised oil pump provides oil, also, for its own pressure gland.

It will be appreciated that the final oil temperature before entering the desulphurising tower is of great importance, in order to ensure the maximum absorptive capacity of the oil in the tower. The theoretical minimum of oil required for complete absorption of organic sulphur from gas in an ideal washer is given by the oil to gas flow ratio such that $qk=1$, where q is the ratio of volume of liquid to the volume of gas passing through the washer in unit time and k is the non-dimensional partition factor, defined as the ratio of the concentration of the solute in the solution to the concentration in the gas. As it is impossible to achieve theoretical design with towers, it is recommended that the oil flow be such that $qk=1.3$ and, to maintain this ratio, it is noted that the oil temperature has a marked effect on the partition factor.

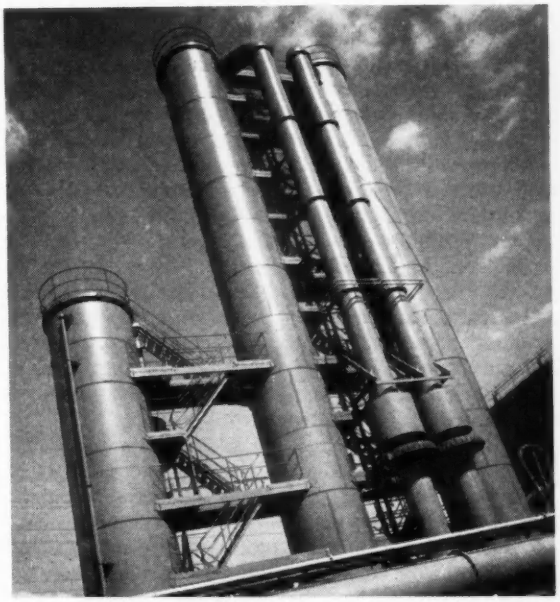
TABLE 2

Temp. of oil °C	Partition factor	Oil gal. per 1,000 cu. ft. of gas to give :—		
		qk 1·1	qk 1·2	qk 1·3
15·5°	335	20·5	22·4	24·2
21°	275	25·0	27·2	29·5
26·5°	227	30·2	32·9	35·6
32°	191	36·0	39·2	42·5

A further practical consideration connected with the oil temperature and, incidentally, in opposition to the above, is the effect of cold oil on gas at a temperature of approximately 30°C., as is the case of the plant under consideration. The temperature of the gas entering the desulphurising tower is largely influenced by the oxide purification conditions, live steam admission, etc. To reduce the amount of condensate deposited in the desulphurising tower, it is the normal practice to maintain the oil temperature at the tower inlet at 2°C. above the gas temperature. Although provision is made for draining the water off the benzolised oil tanks, an excessive quantity can lead to water being pumped into the still with the benzolised oil which condition gives rise to 'bumping' in the still. It should be noted that the practice of maintaining the oil temperature above the gas temperature is far more critical if oil washing is carried out on foul gas since H_2S and NH_3 can be absorbed by the oil and then liberated in the still, giving rise to excessive corrosion.

The process steam used for stripping the oil is saturated pass out steam from turbo alternators at 20 p.s.i. In order that the steam requirements of the plant are reduced to the minimum which incidentally, assist in the elimination of sludge formation, the still is operated under constant vacuum conditions. The vapours leaving the top of the still are condensed by three water cooled shell and tube type condensers before passing to the vacuum pump.

The constant vacuum conditions in the still are maintained by an Aquair vacuum pump of phosphor bronze construction, which discharges the water/benzole mixture into a pumping tank. The gland sealing fluid for the pump is provided by an external water tank, while the sealing fluid for the pump gravitates from the high level receiving tank. The purpose of the pumping tank is to allow for the differences in the pumping capacities of the vacuum and booster pumps. In order that the pumping tank is maintained at constant level, a by-pass from the outlet booster pump is fed back into the tank, the flow being controlled by a ball valve. To circulate the benzole through the high level receiving tank and the benzole/water separator to the daily make storage tank, a booster pump is mounted on an extension of the vacuum pump bedplate and is driven by a common driving shaft from the vacuum pump electric motor.



A close up view of the benzole scrubbers. The naphthalene scrubber is on the left. (By courtesy of W. C. Holmes & Co., Ltd.)

The high level receiving tank is situated above the pumping units and is fitted with a water cooling coil to minimise the effect of the heat of compression. A connection from the base of the tank supplies the sealing fluid to the vacuum pump. The benzole/water mixture flows to a separator from which the condensate flows to an oil separator sump and then to drain, the benzole running to the daily make tank. To avoid explosive mixtures forming in any of the vessels all storage, pumping, receiving tanks and separators are maintained under gas pressure. Wherever necessary the component parts and interconnecting pipe work, such as still, heat exchangers, etc., are lagged with 85% magnesia and waterproofed with emulsified asphalt. The cooling water used on the plant is supplied from a closed circuit, cooling being carried out in an induced draught cooling tower and river water is used to maintain sufficient water in circulation. The requirements for the plant are normally 30,000 gal. per hour at a temperature not exceeding 68°F. Dominating the motor room is an impressive instrument panel on which the pressure and flow re-

corders are positioned in their correct location on a line diagram of the plant.

Steam flow requirements for the plant include instruments for steam for preheating and stripping. A pressure recorder is installed on the steam supply line entering the still. Dial thermometers are situated throughout the plant at useful points and, once the plant is set to suit a particular type of condition, no further alterations need be made save compensation for fluctuating steam pressure. The gas flow through the tower is recorded by means of an Arkon flow meter, situated in the tower purifier installation. For the purpose of these particular tests services were laid from the inlet and outlet gas streams of the desulphurising tower to the calorimeter room and Fairweather calorimeters recorded the calorific value of the gas entering and leaving the plant.

Operational performance. Although considerable work has been carried out by many leading engineers on the computation of formulæ for the operational control of mass transfer plant, the degree of control needed, in our particular case, to give working conditions conducive with efficient sulphur removal, is of a more simple nature involving chemical tests from which an assessment of the plant conditions can be made very rapidly. At the start of this particular series of tests the plant was run with minimum oil flow, *i.e.*, 3,500 gal. per hour. Records were kept of the gas throughput of the tower and calorific value readings at both inlet and outlet. Temperature and pressure conditions throughout the plant were recorded and samples were taken of benzolised oil, debenzolised oil and benzole. The sulphur content of the inlet and outlet gas was determined and routine analysis of the oils and benzole carried out. The steam requirements for this stage of the test was not easily attained. The steam flow included steam for stripping and preheating. The degree of preheat was controlled by the automatic steam valve and the steam to still was set by hand to give a set flow which would adequately strip the oil without excessive steam waste. It has already been explained that 'steam to still' requirements are indicated by the steam pressure and the percentage of distillate below 200°C. is used as an indication of efficient stripping of the oil.

The steam pressure was initially set at 14 p.s.i. and subsequent tests on the debenzolised oil indicated that maximum stripping was being achieved. The amount in excess of minimum requirements, however, was not determined and it was noted that the next increase in steam pressure was not needed until the oil flow was increased to 10,000 gal. per hour. When a series of confirmatory tests had been obtained on minimum oil flow, the oil rate was increased to 5,000 gal. per hour and all previous samples and readings were taken again. Throughout the period, the oil flow was progressively increased until maximum flow was achieved and a record kept of all observations and alterations.

An ideal oil for stripping has yet to be found, but its properties are not in doubt. There should be no sludge formation whatsoever, the specific gravity should be such that the possibility of an emulsion forming with water should be non-existent, the viscosity should be so low that heat transfer should be at a maximum and pumping troubles at a minimum; the boiling point should be high and the distillation range narrow, so as to minimise the amount of oil lost into the benzole, and the absorptive capacity very high. It will be seen from Table 3 that the oil in use at Car House compares favourably with these properties. Unfortunately, we have no first hand information either to viscosity or absorptive capacity but, according to Hutchison, an oil of this type has a viscosity of between 3 and 5 centipoises and a partition factor for carbon disulphide of between 230 and 300. The distillation drop point is about 180°C. so that all the constituents may be considered as high boiling and the distillation range narrow. There have been no sludging troubles since the plant began operation, and it is assumed that the oil is satisfactory from this point of view, although fresh oil is added frequently to make up for normal working losses and considerable quantities added when the carburetted water gas plants are in operation.

TABLE 3

Gas oil quality
(Average for 18 months)

Specific gravity at 60°F	0·831
Flast point	160°F
Carbon residue (Ramsbottom test)	0·07%
Distillation range :	
190 to 250°C	35·0%
below 33°C	95·0%
Specific gravity of distillation :	
Residue at 60°F	0.887
Pour point. Less than	20°F
Composition :	
Unsaturated hydrocarbons	3·3%
aromatic hydrocarbons	19·1%
naphthenes	6·0%
paraffins	71·6%

Table 4 shows that the total steam consumption does not bear a *pro rata* relationship to the oil flow and the reason is thought to be due to: (a) In the early stages of the tests excessive steam to still, (b) variations, due to changes in oil flow, in the benzolised oil temperature leaving the oil to oil heat exchanger, and (c) absence of fine control when resetting the steam pressure.

TABLE 4

Oil flow gal. per hour	Stripping and preheating steam lb. per hour	Lb. steam per gal. of oil
3,500	4,500	1·29
5,000	5,100	1·02
6,000	5,300	0·89
8,000	5,500	0·69
10,000	6,000	0.60
12,500	7,100	0·57
15,000	7,800	0·52
15,500	8,100	0·52

Further tables in the paper showed that, if benzole extraction was the sole concern of the plant, then only under conditions of excessive gas throughput would the need arise for increasing the oil flow above the minimum. With sulphur extraction, however, the oil flow is more critical.

Total sulphur. Since we are concerned only with the total organic sulphur present in gas, for the purpose of these tests, the total sulphur in benzole only was determined. For sale purposes, carbon disulphide is also determined and thiophen calculated as being the difference between the two. Some typical monthly analyses

are shown below in Table 5, the total sulphur, carbon disulphide and thiophen being shown as a percentage of once-run benzole.

TABLE 5

Sp.Gr.@ 60°F	% @ 100°C	% @ 160°C	Sp. Gr. of once-run benzole @ 60°F	% CS_2	% Thio-phen	% Total S
0·858	60·0	88·0	0·859	0·85	0·50	1·35
0·856	58·0	88·0	0·857	1·02	0·40	1·42
0·855	56·0	86·5	0·857	0·82	0·46	1·28
0·856	56·0	85·5	0·857	0·78	0·55	1·33
0·858	55·5	85·0	0·859	0·76	0·54	1·30
0·857	59·0	85·5	0·860	0·84	0·44	1·28

Points to note here are that there is no naphtha present in the benzole, the specific gravity of the once-run is very close to that of the original crude benzole and that only the carbon disulphide and total sulphur are determined experimentally, the thiophen content being the sum total of all other sulphur compounds, not affected by preliminary treatment with 10% KOH. The once-run benzole is washed with caustic and then with water and a portion of sample diluted with alcohol and burned on a wick in a Davidson type total sulphur apparatus. Air cleaned by passing through scrubbing towers moistened with sulphuric acid and then caustic is used for combustion purposes and the products of combustion absorbed in neutral hydrogen peroxide. After a suitable quantity of solution has been burnt, the acidity of the solution is determined with standard alkali and hence the sulphur content of the benzole calculated.

Sulphur in gas. For these tests, the gas was burned in an apparatus of similar design to that used for sulphur in benzole. The arrangement is to pass the gas through an oxide tower to remove any traces of H_2S, through a wet meter and then burn it in a stream of clean air and absorb the products of combustion in neutral hydrogen peroxide. No attempt has been made to differentiate between the various organic sulphur compounds, nor did we take into account the presence of oxides of nitrogen, which produce nitric acid on oxidation. The latter error may be overcome by precipitating the sulphates as barium sulphate, but the error is probably so small that, for all but the most accurate work, it may be ignored.

If the percentage of sulphur extracted is plotted against the ratio of gas flow in cu.ft. per hour to the oil flow in gal. per hour, *i.e.*, the removal efficiency, it can be seen that the sulphur extraction does not increase in proportion to the amount of oil used for scrubbing. In addition to this, if the oil is not properly stripped in the distillation unit, sulphur compounds will remain in solution in the oil and prevent sulphur compounds from being extracted from the gas, because a state of equilibrium will be reached by the absorption of less sulphur compounds from the gas. We attribute some of the lower percentage extractions to this cause. As our experimenting is likely to continue for some time, we hope to investigate this, and any other anomolous results, further.

The temperature of the debenzolised oil entering the scrubber is of paramount importance inasmuch as the position of equilibrium is affected to a high degree for comparatively small changes of temperature. A higher temperature will obviously increase the vapour pressure of the solute and will effect the lower boiling constituents much more than the higher, *i.e.*, carbon disulphide and thiophen respectively. The boiling points of these two compounds are 46°C. and 84°C. so that, for carbon disulphide at any rate, the oil temperature is not too far removed from its boiling point. As thiophen has a similar boiling point and, therefore, a similar vapour pressure to that of benzene, we assume that it will be affected similarly. Tests using the St. Clair Deville apparatus, cooled in a solid carbon dioxide/acetone mixture showed that the benzole extraction, even at low oil flow rates, was in all cases, well over the 92% mark. As carbon disulphide is present in gas in the greatest proportion between 50-60%, it follows that, to get the lowest possible organic sulphur, conditions will have to be adjusted to suit this compound. High oil rates at low temperatures are desirable so that maximum absorption can take place, but limits must be imposed for economical reasons, namely, the size of plant, amount of heat available, and the amount of cooling applied to the oil and gas.

The effect of benzole extraction on gas output can be studied by considering the reduction in calorific value due to oil washing. The average drop in c.v. throughout the tests was found to be 28 B.t.u. per cu.ft. and assuming 25 mill. cu.ft. per day gas throughput then the thermal reduction would be 7,000 therms per day. With the oiling rate on the carburetted water gas plant set to give a c.v. of 500 B.t.u., then the variable factor in gas output, affected by benzole extraction, is the quantity of producer gas added for dilution to bring the c.v. of the coal gas and coke oven gas down to the declared figure of 500 B.t.u. per cu.ft. Assuming the c.v. of producer gas to be 120 B.t.u. per cu.ft. and that the reduction in gas volume, due to the extraction of the hydrocarbons to be negligible, then the amount of producer gas which can be added when the plant is shut down is calculated as 1.85 mill. cu.ft. per day. This represents an increase of 7.4% of the gas output which, in cases of maximum demand, is a substantial increase when gas volume is the main consideration.

Sulphur balance. There was very close agreement between the sulphur removed from the gas and the sulphur present in the crude benzole, as seen from Table 6.

TABLE 6

% S in Crude Benzole	Benzole made gal./day	Wt. of S Lb.	Gas flow mill. cu. ft./day	S extracted grains/ 100 cu.ft.	Wt. of S Lb.
1·07	2,000	185	9·8	12·6	177
0·98	1,800	149	8·1	11·6	134
0·95	1,800	145	8·4	12·3	147

It will be seen that the plant behaves in a steady well controlled manner and it is possible, even with fluctuations in gas flow and organic sulphur content, to maintain the organic sulphur content below 10-grains per 100 cu.ft. Although the inlet sulphur content at Car House is reasonably steady, we feel that a gas to oil ratio of about 50 cu.ft. gas per gal. of oil will easily maintain the sulphur content at a satisfactory level and that, should this level increase due to excessive sulphur at the inlet scrubbers, the plant is sufficiently flexible to cope with these contingencies.

SODIUM SILICATE *as a Corrosion Inhibitor in Gasholders*

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AN investigation has been carried out to study the effect of the addition of sodium silicate as a corrosion inhibitor in the water contained in gasholders. Over a period of 13 months steel test panels were hung in a silicate treated and untreated holder and removed at intervals for examination. The final results, while not being wholly conclusive, suggest that the life of a holder would be increased materially by silicate dosage. In the case of unpainted steel an increase in life of as much as 30% to 60% was indicated. Furthermore, it was noticed that the silicate treatment markedly reduced staining of the paint-work by red water. This may, in some cases, be more significant than actual corrosion inhibition and further work is to be carried out on this aspect.

Effective in Water

Information received from America in 1955 suggested that the addition of sodium silicate to the extent of 80 ppm. as SiO_2 is an effective corrosion inhibitor in gasholder water. Based on this report laboratory tests were carried out and the results obtained indicated that corrosion could be expected to be reduced, but because of the difficulty of reproducing working conditions in the laboratory the findings were not considered to be entirely conclusive. Accordingly, a full scale trial was arranged in collaboration with the North Western Gas Board. Two holders were selected for the trial, each of 600,000 cu.ft. gas capacity. Both holders were in good condition, newly painted and comparable in all important respects. It was decided to add the sodium silicate to No. 1 holder for test purposes and to use No. 2 holder as a control.

The water tank of the holder used for the test had a capacity of 870,000 gal. Pyramid brand sodium silicate No. 1 containing 29% silica as SiO_2 was used as the inhibitor and an initial charge of 30 cwt. of silicate was added. This is equivalent to a concentration of 112 ppm. of silica as SiO_2. To help ensure good mixing the silicate was added in dilute form. A small tank was placed by the holder and connected to it by temporary pipe-lines through a pump. The diluted silicate was added in small quantities from the side tank over a period of two or three days in the middle of March, 1956. Despite this precaution it seems that the silicate must have sunk to the bottom of the tank, for a month later the surface water contained only a nominal amount of silica. By the beginning of June, however, a surface concentration of 50 ppm. had been reached and a month later this had risen to 75 ppm. As this silica concentration was rather lower than expected—possibly due to reaction and adsorption on the sludge no doubt present in the holder—arrangements were made to add a further 10 cwt. silicate at the end of July, 1956. Apart from this other refresher charges were made throughout the cou of the trial which lasted until August, 1957, a total peri of 13 months. At the end of this time the silica conce tration in the test tank was still 80 ppm.

Both holders consisted of two lifts and on July 6, 19 12 test plates were hung in each holder, six in the ta and six in the cup. Since it was the inhibitive effect the silicate which was under investigation, it seem better to use unpainted steel for the test plates so as study the effect which would occur when the paint co ing in the holder should break down. These plates we of unpainted mild steel 12 in. by 12 in. by $\frac{1}{16}$ in., all c from the same sheet, in the as-rolled condition, solve cleaned and suspended so as to be about half immers in the water. Each plate was coded and careful weighed and measured for thickness before test. It w the intention to remove certain of these plates at interva to study the course of corrosion. The silicate concentr tion in the water was determined colorimetrically by t ammonium molybdate method and is expressed as pp of silica as SiO_2.

When a set of plates was removed, each was fir cleaned by wirebrushing, followed by a short immersi in inhibited acid; a blank experiment showed that n attack occurred in cleaning. The plates were the weighed to find the total loss which had occurred an measured accurately at a large number of points t determine the thickness of metal which had bee corroded away. In order to measure the depth of an pits, one anvil of the micrometer was a needle. Observa tion of the corroded test plates showed three distinc zones of corrosion: Above the water line where th main corrosion had occurred, a narrow band about 1 i wide at the water line, and the bottom zone which wa fully immersed. The area of each zone was measured It was found that within each zone the penetration o the metal was about the same in that area.

Maximum Extent

It was apparent that the loss in weight measuremen was of little direct use as it averaged the results for th three different zones. Of greater importance was th maximum extent of the penetration which had occurred as the limiting factor in the life of a gasholder plate is its perforation. For each plate, and for each zone on that plate, the average loss in thickness and also the maxi mum penetration which tended to occur in pits was measured. Such measurements are prone to a wid margin of error, but a check on these figures was made by calculating the loss of weight for each plate on the

basis of the area of each zone and the average loss in thickness of that zone; this calculated figure could be compared with the actual loss in weight. Good agreement was, in fact, found, suggesting that the figures for loss in thickness, which was the main criterion, were accurate.

Results. (a) At three months: Four plates, one each from cup and tank of control and test holder were removed after three months. The corrosion pattern was similar on all four plates, with three distinct zones of attack which were quite clearly defined. Below the water line on the bottom zone there was very little corrosion, the metal remaining smooth. At the water line was a zone which was essentially smooth but with slight pits. Above this, right up to the top of the plate was heavily corroded, the surface being rough and badly pitted. The results are shown in Table 1.

TABLE 1. Corrosion after three months

	Penetration in ·001 in. (*a*)						Wt. loss (*a*)	
	Top zone		Water-line zone		Bottom zone		Calculated	Observed
	Max.	Av.	Max.	Av.	Max.	Av.		
Treated Tank ..	15	12	5	3	2	1	71 g.	63 g.
Control Tank ..	17	12	5	3	2	1	85 g.	69 g.
Treated Cup ..	6	4	4	2	2	1	31 g.	29 g.
Control Cup ..	10	8	3	2	2	1	93 g. (*b*)	88 g. (*b*)

a. The figures in each case refer to one plate only.
b. The plates in the control cup were immersed for about 6 in. instead of 8 in. for the others ; Therefore, the area heavily corroded above the water-line was greater—hence the high weight loss.

It may be concluded from this that the silicate addition at this stage had had very little effect on the corrosion rate but, if anything, it may have been slightly beneficial. Also at this stage there were signs that the rust staining of the paintwork by the water had been reduced.

(b) At six months: Eight more plates were removed three months later, two from each location. After a total test period of six months, the results are shown in Table 2:—

TABLE 2. Corrosion after six months

	Penetration in ·001 in. (*a*)						Wt. loss (*a*)	
	Top zone		Water-line zone		Bottom zone		Calculated	Observed
	Max.	Av.	Max.	Av.	Max.	Av.		
Treated Tank ..	29	21	5	3	2	1	119 g.	118 g.
Control Tank ..	28	18	6	3	2	1	106 g.	117 g.
Treated Cup ..	21	15	8	4	4	2	109 g.	113 g.
Control Cup ..	27	21	16	7	6	4	215 g. (*b*)	205 g. (*b*)

a. All results are the average of two test plates.
b. The plates in the control cup were immersed for about 6 in. instead of 8in. for the others ; therefore, the area heavily corroded above the water-line was greater—hence the high weight loss.

These results indicated that there was no benefit from the inhibitor in the test tank but, rather surprisingly, there was possibly some slight benefit in the cup. The results did not, however, suggest that the silicate addition had been worthwhile.

(c) At 13 months: Since the six months' test had indicated that there was very little corrosion inhibition by the silicate, it was decided at 13 months to remove all three plates remaining at each location with the object of securing results, in triplicate, which should give a more accurate assessment. The results obtained are shown in Table 3.

TABLE 3. Corrosion after 13 months.

	Penetration in ·001 in. (*b*)						Loss in Wt. (*b*)	
	Top zone		Water-line zone		Bottom zone		Calculated	Observed
	Max.	Av.	Max.	Av.	Max.	Av.		
Treated Tank ..	33	25	5	2	2	1	178 g.	188 g.
Control Tank ..	56(*a*)	43	16	7	3	2	270 g.	273 g.
Treated Cup ..	39	32	6	4	4	3	204 g.	211 g.
Control Cup ..	49	43	26	23	8	5	396 g. (*c*)	373 g. (*c*)

a. Two of the three test plates in the control tank were corroded right through.
b. All results are the average of three test plates.
c. The plates in the control cup were immersed for about 6 in. instead of 8 in. for the others ; therefore, the area heavily corroded above the water-line was greater—hence the high weight loss.

At three months there was no significant difference in the penetration of the test and control panels, the maximum penetration being .015 in. and .017 in. in the tank, .006 in. and .010 in the cup, for test and control panels respectively. At six months there was still little difference, the figures being .029 in. and .028 in. in the tank, .021 in. and .027 in. in the cup, in test and control panels respectively. At 13 months the difference was very marked. In the tank the figures for maximum penetration were .033 in. and .056 in. in the test and control panels respectively, while in the cup they were .039 in. and .049 in. respectively. This superiority of the test plates applies whether it is the maximum or average penetration, water line zone or bottom zones which are compared, but the case of the most heavily corroded zone has been taken in the example given above.

Based on the figures for penetration the probable life of an unpainted $\frac{1}{4}$ in. steel plate would be seven and a half years in the test holder tank against four and a half years in the control tank (an improvement of over 60%); or of six and a half years and five years respectively in the cups (an improvement of 30%). The fact that the inhibition is less in the cup than in the tank is not surprising because of the dilution which occurs from rain water and because of the few occasions when the water in the cup becomes re-charged from the tank. Comparing the penetration at periods of three, six and 13 months, it is apparent that the corrosion which had been occurring at a steady and nearly equal rate for six months in the test and control tanks ceased suddenly after six months in the test tank, but went on steadily in the control tank. There is no obvious explanation of this, but the validity of the results is supported by the similarity of the measurements found on the plates removed from each location, also by the very close agreement between the loss in weight calculated from the penetration and that found experimentally.

The degree of benefit which has been obtained as the result of the addition of 80 ppm. of silica as SiO_2 is somewhat difficult to assess in terms of figures. However, there seems to be no doubt that the sodium silicate has assisted in reducing corrosion of the metal. Clear evidence of this is the fact that of the six plates removed from the tanks after 13-months, two were corroded right through over an area of several sq. in. above the water-line and both these plates were from the control tank. No case of perforation occurred in the silicated test tank. A reduction in the rate of corrosion was not observed immediately the sodium silicate was added, and a period of over six months was necessary before any appreciable benefit was apparent. However, the results of the 13 month test suggest that the life of unpainted steel would be appreciably lengthened.

Investigation into the composition of ammoniacal liquor—

II: A SURVEY OF EFFLUENTS PRODUCED IN THE GAS AND COKING INDUSTRIES

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THE paper is concerned mainly with the determination of the concentration of different phenols in ammoniacal liquors from various sources. The methods generally accepted for their determination, as described by Key ('Gasworks Effluents and Ammonia,' second edition, I.G.E., 1956) are not entirely satisfactory. In these, phenolic compounds are divided into two groups, 'monohydric phenols' and their homologues, and 'higher tar acids' which are polyhydric phenols consisting of catechol, resorcinol and quinol and their homologues.

The former group is determined by estimating the bromine absorption value, and the latter by taking the alkaline oxygen absorption figure. Both determinations make assumptions which are only approximately valid.

By adapting the technique of chromatography, the authors have been able to carry out much more satisfactory analyses which give reasonably accurate figures for the concentration of the individual phenolic components of both groups. By this method, the phenols are separated directly from 1 ml. of an acidified liquor by column liquid—liquid partition chromatography, the concentrations being measured by ultraviolet spectrophotometry. Many phenols are completely resolved, though some appear in mixtures of a few isomers.

For a more rapid determination of total phenols in ammoniacal liquors, a method is suggested that involves only measurement of the ultraviolet absorption of a suitably diluted sample of acidified liquor before and after extraction with ether. Total organic bases may also be obtained in a similar manner.

It has been shown that, in all liquors, the simpler molecules are present in the greater proportion, concentrations decreasing with increase in molecular weight, so that phenols with three or more carbon atoms in side chains are insignificant in amount. In most liquors, phenols is the major organic constituent and the concentration of monohydric phenols is greater than any group of related dihydric phenols. Frequently, the concentration of homologues are in the order:

monohydric phenols > catechols > resorcinols > quinols.

There is no doubt that the lower-temperature processes result in liquors with the greatest concentration of dihydric phenols. In high-temperature practice, contact of the crude gas with the hot walls of the retort is not sufficiently long in vertical retorts to produce a substantial decrease in the concentrations of phenols in liquors. With horizontal retorts and coke ovens, however, there is greater destruction of such compounds, dihydric phenols being far lower in concentration in liquors from these plants. In this respect, the resorcinols would appear to be a little more stable to thermal decomposition.

Different parts

There is, of course, a difference between the concentration of phenols in liquors from different parts of a plant. Retort house liquors contain a high proportion of dihydric phenols, and the catechols can displace the monohydric phenols as major constituents. In the primary condensers, the dihydric phenols are present in lower concentrations than in retort house liquor, though monohydric phenols are similar in amount. Hot gas electro-detarring gives a condenser liquor that contains very little dihydric phenols, but tends to result in an increased concentration of monohydric phenols. In these liquors, the catechols, which are more volatile than other dihydric phenols, form the greater proportion of such compounds. Except for reductions in the concentrations of the more volatile phenol and *o*-cresol, recovery of ammonia yields a 'spent liquor' in which the concentrations of other phenols are not very different.

If any further investigations are to be considered, it is clear that more analyses should be made of liquors from vertical and horizontal retorts to determine how the different factors of the source of coal carbonised and plant operation affect the liquors. An interesting consideration would be the determination of the constituents of both tar and liquors at a given works and, from the volumes produced, to obtain a balance for the different products.

In a previous investigation into the possibilities of devising a chromatographic method of analysis of the organic constituents of liquid from a continuous vertical retort system the total organic matter was extracted by means of an organic solvent, and subdivided into organic acids, phenols, bases, and neutral oils by alkali and acid extractions. Of these groups of compounds, the phenols were present in the greatest concentration, forming about 95% of the total organic matter.

By fractional distillation of the phenols, followed by analysis of the monohydric phenol fractions by infra-red spectrophotometry, and of the dihydric phenol fractions by ultraviolet spectrophotometry after further separation involving chromatography, it was possible to determine the concentrations in the original liquor of some 35 phenols.

It was obvious, however, that the direct application of this method of analysis to a series of liquors would be a lengthy operation. Apart from which, quantitative recovery of all compounds was not possible.

In the first place, since the phenols were present in small concentration, it was necessary to extract them from a large volume of liquor to produce an amount of material sufficient for distillation; thorough extraction was necessary to recover the more water-soluble phenols. Secondly, it was not possible to obtain a clear separation of one group of homologues from another by alkali and acid extractions. Finally, the most serious factor was the resinification of dihydric phenols during fractional distillation and, to a smaller extent, during extraction with alkaline solutions, leading to production of a phenolic pitch.

Such considerations made essential a

different approach to the problem. Experience with liquid—liquid chromatography as applied to dihydric phenol distillate fractions indicated that this might be extended to the complete analysis of the phenols in a liquor, and, with some minor reservations, this can now be claimed to have been achieved.

Considerable evaporation

Paper chromatography had given better separations of dihydric phenols than column chromatography, but it was difficult to extend the former technique to include monohydric phenols since there was considerable evaporation of these compounds during development. Reverting to column partition chromatography, therefore, it was found that substantial modifications of the existing method were required for extension of the analysis. The procedure had involved the use of a column of silica gel to support a stationary phase of water, the dihydric phenol fraction being added to the top of the column and the components separated by elution with benzene, to which butanol was added in gradually increasing amounts in order to elute the more water-soluble phenols. Concentrations of material in the eluate were determined by measurement of the ultraviolet absorption at the wavelength of maximum absorption for the compound concerned.

In modifying the procedure, for better quantitative determinations, it was necessary to replace benzene as eluent, since the ultraviolet absorption bands of benzene obscured those of a number of phenols. *Cyclo*hexane has been found to be a more satisfactory eluent in that it has no ultraviolet absorption in the region required in the analysis, while the resolution of dihydric phenols was similar to that obtained with benzene. Improved separations have been achieved by substituting Celite 535 for silica gel as support for the stationary phase, the latter now being an acid solution to minimise oxidation of dihydric phenols and to prevent the elution of organic bases when a liquor is entered onto the column. With such a column, it is possible to resolve the mixture of dihydric phenols that is found in a liquor.

Of the monohydric phenols, only phenol separates as a discrete band before the dihydric phenols, but all the other monohydric phenols are eluted together in a band showing two maxima. These monohydric phenols can, however, be separated with a stationary phase of high pH_2. White[1] has suggested the use of sodium silicate solutions for this purpose, and satisfactory separations have been made in the present work using different concentrations of sodium silicate solutions for the resolution of cresols and of higher-boiling monohydric phenols.

The next consideration was the quantitative transfer of phenols from the liquor to the column; and, in order to eliminate errors of extraction, chemical separation and distillation, it was decided to add the liquor directly to the chromatographic column. Calculation indicated that the concentrations of phenols in the eluate from such a column would be of the right order from 1 ml of liquor containing above 1g/l. of total phenols. Mixing this quantity of acidified liquor with 2g of Celite produced a material that could be added quantitatively to the top of the column and be of the same composition as that of the rest of the column. At this point, no separation of other groups of homologues from the phenols had been made. Neutral oils were, however, eluted at the beginning of the chromatogram and could, if necessary, be distinguished by their ultraviolet spectra. Aliphatic carboxylic acids did not interfere, since they have no ultraviolet absorption spectra, while organic bases would mainly be held on the acidic column, or, if eluted, have ultraviolet spectra different from those of the phenols.

Complete resolution of all the phenols previously identified in the vertical retort liquor cannot be obtained in the procedure employed at present, but all those of major importance may be determined. This includes all the parent compounds (phenol, catechol, resorcinol and quinol) and their methyl homologues (the exception being 2- and 5-methylresorcinols, which are eluted simultaneously); but these compounds together form about 90% of the total phenols, the total concentrations of all isomers having eight carbon atoms being substantially less than those with seven, even though a greater number of compounds are involved. These higher homologues are, however, resolved into related groups containing at the most only a few compounds, and it is considered that the analysis is sufficiently detailed for most purposes for which it should be required.

A brief account is given of the apparatus and analytical procedure, and tables are given showing the composition of liquors obtained from low temperature carbonisation, continuous and intermittent vertical retorts, horizonal retorts, coke ovens and Lurgi plant. Comparisons are made showing the differences in composition of the liquor obtained from different positions in the flow of the gas stream.

[1] 'The Theory and Practice of Liquid—Liquid Partition Chromatography of Phenols.' White D., and Vaughan, G. A. *Anal. Chem. Acta*, 1957, **16**, 439.

DISCUSSION

Dr. H. Hollings: There is no doubt in my mind that all this very precise scientific work which has been conducted over such a period by the gas industry is very well worth while, because I am sure that the gas industry has built up a tremendous goodwill with sewerage authorities and river boards up and down the country as a result of this very careful approach towards a solution of its own problems. Furthermore, the efforts of the carbonising industries as a whole form no small part of the British industry effort to arrive at the time when we shall have much cleaner rivers in this country. Only a few days ago I heard a German authority explaining that some of the Continental water authorities point to British industry as an example of what their own national industries might attempt to follow.

What are the main lines of work upon which the Research Committee is engaged at Leeds? May I try to remind you of one or two of them? At the Leeds sewage works we have now proceeding quite an extensive series of experiments in which lined and unlined continuous vertical retort liquor and a coke oven liquor are being treated in a mixture with domestic sewage on biological filters. There is a very considerable number of filters involved in this programme, and the important feature of the work is that it is all being planned under expert statistical advice, so that we hope in due course we shall be able to arrive at a proper correlation between small-scale laboratory work and large filters working in practice. Then in Table 4 of the paper you have before you several analyses of various liquors derived from the Leamington gasworks, and I would just remind you that at one of the smaller sewage works of the Coventry Corporation waste liquors are being treated, again in a mixture with domestic sewage. The main practical objective of that work is to establish for the gas industry more precisely just what is the effect of the segregation of retort house liquor and just what is the separate effect of the installation with the hot gas detarring process. That work is in a very advanced stage, and I hope that very shortly we shall have a final publication upon it.

Then in the laboratories in the University experiments are proceeding on various coke oven liquors which are being treated with diffused air without a mixture of any domestic sewage. A very informative and very interesting paper by Mr. Catchpole was published upon that work in this room but before the Iron and Steel Institute just a year ago.

The future

There, then, are three main lines of work proceeding towards severely practical objectives at the present moment. What of the future? One or two of us feel that perhaps this work has proceeded to the point now at which we might revise our programme. Perhaps there might be some redeployment of our forces. My own opinion is that Mr. Catchpole's work with these liquors being treated by the diffused air process, in the absence of any sewage addition, has proceeded just about as far as it can proceed usefully on the laboratory scale. During the past 12 months we have looked around for some suitable coke oven site at which we might transfer this work on a larger scale, but so far, I regret to say, without success.

A second line of new work obviously suggests itself when you consider what is being done at the Leeds sewage works. It has been pointed out from more than one

area gas board that in other parts of the country the activated sludge process of sewage treatment is more favoured by the sewage engineers than the biological filter, and therefore it is suggested—and I agree—that we ought to be carrying out experiments in which the different waste liquors are treated with sewage in the activated sludge process. The real purpose, whether it be an experiment on the biological filter or whether it be an experiment in the activated sludge tank, is to try and answer the question which I know is very important in the minds of many gas engineers: How much extra sewage plant capacity, filter or big tank is necessary when the ammonia waste liquors are mixed with the sewage?

Investigations

Then, while these large-scale plant experiments are proceeding, there are, of course, quite a number of investigations which should be continued much further in the laboratory. I think that we have reached the stage at which purely on the laboratory scale we shall have to pay more attention to the possible methods of treatment of those components of the liquor which are not amenable to biological oxidation. In this field much pioneer work has been done at the Fulham laboratory, and if you will allow me, I would, on behalf of my colleagues on the Research Committee and on behalf of the staff at Leeds, like to acknowledge our indebtedness to Mr. Badger and his colleagues for all the help they have given us, particularly to Mr. Badger for his very useful contributions on so many occasions to our discussions in Committee.

Another possibility is, of course, that the residual components might be oxidised, or alternatively there are facilities of adsorbing some of the substances on mineral clay.

But all these are just possibilities which still need a good deal of further study on the laboratory scale. I do not suggest, however, that there is any quick or inexpensive answer to the problem. I just thought it appropriate to try and indicate some of the lines on which work is proceeding.

Mr. G. Boreham, London Research Station: The obvious method of separating the phenols before analysis is by some form of chromatography. Nowadays it would seem that gas chromatography is the answer for most types of analysis, but it does not give us the separation required here—at least, not at present—although many workers all over the country are carrying out research on this project. It has been calculated that to separate m- and p- cresols a column in excess of 200,000 theoretical plates would be required. Added to this, gas chromatography cannot as yet operate on very dilute components in aqueous solution.

My only real criticism and surprise with the method of analysis used is that a column chromatographic method is used for the analysis of dihydric phenols in a liquor. I realise that the aim is to have a speedy analysis, but if we refer back to an earlier paper, G.C.24, we can at once see that if paper chromatography is used for this separation then a very much better separation is achieved—all the monohydric phenols with the exception of the indenols and 1- and β-naphthols appear at the solvent front (using benzene-acetic acid-water in the ratio 3:3:1) and an almost complete separation of dihydrics (and also main trihydric phenols) is accomplished.

As Dr. Hollings has also said, at the London Research Station a pilot plant study of the bacterial oxidation of phenol is in progress, and a complete picture of all the phenols in all liquors to be treated is necessary. Further to this, active research is being carried out on tracing the oxidation path of phenols by a pure stream of organisms isolated from the mixed bacteria. Hence it is necessary to be able to pinpoint any particular phenol, and we feel that this is best achieved by means of paper chromatography. Accordingly, the method of analysis in G.C.24 has been developed to give a quantitative picture of dihydrics present. After their separation, by development in the solvent, has been achieved, the phenols are eluted from the paper and estimated as before by their absorption in the ultra-violet.

My own experience with column-chromatography has led to two conclusions: (1) That it is very important to ensure that the Celite packing is free from iron and free from any form of fatty material. If the iron is not all removed, then phenols are easily adsorbed and are not eluted; the fatty material appears in the eluant and gives false spectra and readings at some point in the separation. (2) It is very desirable to cover a fairly wide range of wavelength absorption for each fraction to ensure that only the phenol is being measured. Hence a recording ultra-violet, though an expensive luxury, is very helpful, otherwise analysis along these lines will take a long time.

There is one further minor criticism. The authors state that aliphatic carboxylic acids do not interfere with the analysis. It is true that fatty acids such as formic, acetic, etc., do not interfere, but acids such as oxalic, pyruric and laevulic will give a background absorption.

Quick method

The authors have done well to formulate a new, quick method for estimating total phenols. Methods now used in control laboratories usually consist of determining monohydric phenols with 4A/P and total phenols with the F/D reagent. Experiments carried out last year have shown that p-substituted phenols do not react with 4 A/P and if they do they do so only very slightly. Meta cresol only gives 70% of the correct response and hence the figure for monohydrics by this colorimetric method is markedly low.

Mr. J. Young, Midland Tar Distillers Ltd.: Dr. Barker and Mr. Hollingworth have made a most useful contribution to the techniques available for the analysis of phenols. We at Midland Tar Distillers have made use of their methods as described at these meetings three years ago, and we regard liquid-liquid chromatography as a most valuable technique, particularly for dihydric phenols. It has some distinct advantages over gas-liquid chromatography. There must be several chemists here who would wish to use these methods and they may like to hear that these rather lengthy determinations do lend themselves to a semi-automatic procedure.

We have modified a Unicam SP 500 spectrophotometer so that it will operate a recorder. The modification is a very simple one and particulars are being published in *The Analyst*. We set the instrument at a fixed wavelength—generally 2,800 Å3—and pass the eluate from the column through a small cell suspended in the normal cell-well. The run generally takes place overnight drawing a graph similar to those in the paper under discussion. It has the advantage of being a continuous record, rather than a graph drawn from the optical densities of discrete fractions, and both quantitative and qualitative analyses can generally be made from the graph alone. If necessary, the eluate is varied continuously by using two fluids. The lower one contains cyclohexane. As eluent from it flows on to the column, it is replaced by a 10% solution of butanol in cyclohexane from the upper flask. Variations in water content are adjusted by passing the eluent through a short silica gel or Celite column before the main one.

This type of semi-automatic procedure will be found useful if determinations are to be made with any regularity. We hope to offer the method for publication shortly.

Impurity peak

A section of the present paper deals with the impurity peak eluted just before 3-methylcatechol. We can confirm the observations made point by point but cannot explain them. We have also had spectra of both the types shown on [p. 50 and 51]. The peak is sometimes needle sharp being eluted in about 2 ml.; on other occasions it is partly resolved and appears as a doublet. I certainly agree that the explanation is not a simple matter of oxidation of dihydric phenols even though they give similar ultra-violet spectra. If no ultra-violet spectrophotometer is available, we have found it possible to titrate eluate fractions directly using a quaternary ammonium hydroxide and an indicator.

Finally, I should like to ask a question. On [p. 5], mention is made of stationary planes of high *p*H for the separation of monohydric phenols. Would the authors say a little more about their experience both with sodium silicate and other alkaline materials?

Dr. R. L. Cooper, British Coke Research Association: The effluent problem of the coking industry has much in common with that of the gas industry, and recognising this fact, the British Coke Research Association is actively supporting the work of the Joint Research Committee of the Gas Council and the University of Leeds in this field and is represented on the Ammonia Effluents Sub-committee.

The situation in the coking industry is possibly more acute than it is in the gas

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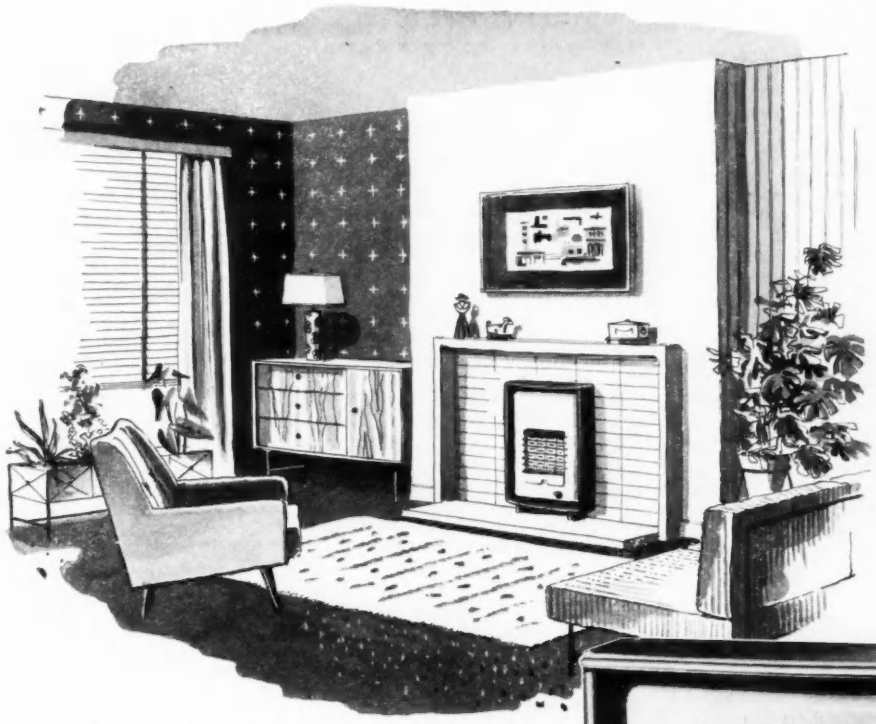

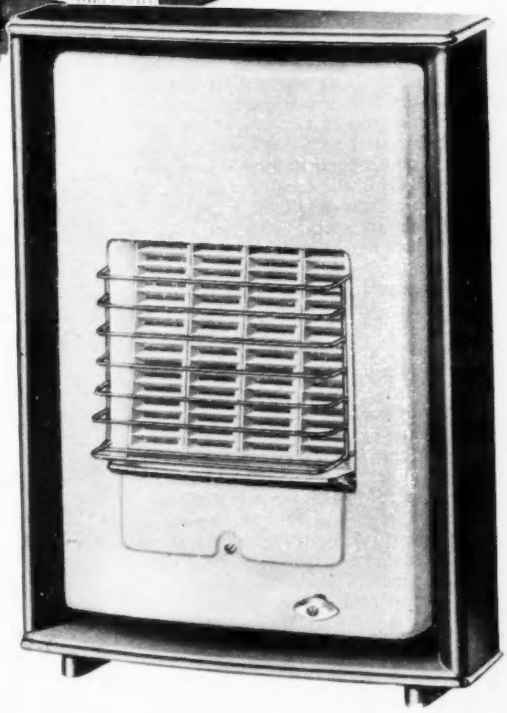

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industry, since few coke oven plants can discharge their effluents to the sewers and some form of complete treatment is required before discharging into rivers. Alternative methods such as disposal to spoil heaps, bore holes or shafts are merely palliatives. The employment of spent liquor in coke quenching leads to undue corrosion of coke cars and surrounding steel work resulting in costly repairs and replacements. The use of the crude liquor as an agricultural fertiliser is unattractive on account of its low nitrogen content.

Hit and miss

If liquors are to be treated efficiently it is obviously of first importance to know their composition and the influence of factors which can be used to effect modifications. Hit and miss methods might work for a particular plant, but only systematic investigation will suffice to solve the varied problems of a whole industry. In the paper under discussion, we have an investigation into a group of substances which represent 90% of the organic content of liquor and which has a profound influence on treatment processes; if allowed to go untreated into a river, on the condition of that river, and possibly of water subsequently used for domestic purposes. The account of this investigation must surely rank high in the literature of analytical chemistry. Until this work, there was no really satisfactory means of analysing such complex mixtures of phenols. We have seen the authors carrying out much necessary fundamental work and steadily progressing from conventional but laborious methods until they have now arrived at a relatively simple and reliable technique. A few years ago I initiated work on phenol analysis in which the compounds were converted to their methyl ethers and then separated chromatographically on alumina and determined spectroscopically. The method was developed and improved by my two colleagues Commins and Lindsay and is published in the literature. Although the method was neat, and only 1 ml. of sample would be required for liquor analyses such as these, it suffered from inherent disadvantages and would not allow the successful resolution of such complex mixtures as the authors of this paper have attempted. I think their method, with suitable modifications, will find a wide application in research and industry in general.

To return to the coke oven effluent problem, it now seems that the treatment of effluents by biological processes on site offers the cheapest satisfactory means of mitigating liquid pollution in the general case, though individual circumstances might well dictate otherwise. Circumstances again will decide which of the alternative methods of biological treatment is most desirable. Assuming it is to be adopted, a knowledge of the phenol content of the liquor will indicate the greatest individual load on the plant. In this connection, we see from p. 30 of the paper that the spent liquor from the ammonia still contains most of the monohydric phenols and virtually the whole of the polyhydric phenols originally present in the crude liquor. The compounds in this latter group appear to interfere with the biological oxidation of other liquor constituents, give rise to coloured complexes as a result of chemical oxidation, interaction with each other and possibly with iron surfaces, and contribute to the residual oxygen demand of the final effluent, thus lowering the efficiency of the process. They also cause difficulties with other methods of treatment and incidentally are disliked by sewage works managers. In view of this, we wish to know for certain how the concentration and relative proportions of these compounds vary from one liquor to another and according to the conditions of carbonisation and type of condensing and scrubbing plant. In the gas industry, we see that hot gas electro-detarring and the separation of retort house liquor from the condenser liquor circuit are being explored as a means of reducing the polyhydric phenol content of the liquor to the still. In the coking industry we wanted to know if any parallel modification of works practice was necessary on this account. Until recently we have never been quite sure how important these compounds might be, although it was assumed that their concentration in crude liquors would be low due to the conditions of thermal cracking in the ovens. Several coke oven crude liquors have now been analysed and the figures are to be found in [Table 7 on p. 35]. I regard these results as the first reliable data on the phenolic content of these liquors. Together with some additional figures which I have been given they cover a wide range of conditions, namely, the carbonisation of coals of high and low rank, both semi-direct and indirect processes of ammonia recovery and a limited variation in the temperature of carbonisation. It will be seen that irrespective of these differences, the polyhydric phenol concentration in the liquor is of the order of 100 to 200 mg. per litre and so there seems to be little reason to modify works practice on this account, unless it be a relatively minor alteration to improve the residual oxygen demand of a treated effluent. Conversely, the results suggest that the spent liquors would be suitable for biological treatment when the inorganic ion content is also taken into consideration.

Now that we have a means of analysing all the major constituents of carbonisation effluents we are in a better position to assess the relative merits of the varied methods of dealing with the effluent problems, according to the prevailing circumstances. We look forward to further improvements in the technique which will enable us to carry out the determination with greater rapidity.

Dr. L. Barker: Mr. Boreham brought out the point about gas chromatography that it cannot compete with our method at present. As regards paper chromatography, we have found that it gives better separation in the paper, although some of the quantitative aspects did not satisfy us very much; but I know that Mr. Boreham has found it satisfactory in his case. I think our main disagreement with the method is the fact that you have need to extract the material from the liquor before you can apply it to the paper.

As regards Mr. Young's remarks, we have seen his apparatus and we do like it. I think there is a lot of donkey work involved in the method as published in our paper, and any automatic procedure which can get over that sort of thing is to be encouraged.

I think that Dr. Cooper did well to bring out the point about forming complex compounds with phenols. It is not a very quantitative method to do it in that way.

In conclusion, I should like to thank those who have contributed for their kind suggestions, and to say that a full reply will be given in writing.

Centuries ago the Chinese used natural gas which was brought by bamboo-tube to a mound of clay. A replica of one of these primitive appliances was shown by Mr. L. W. Andrew, the Director of the Gas Council's Watson House Centre, when he gave the Institution of Gas Engineers' annual Christmas lecture to young people.

Kestner's fifty years of endeavour

To commemorate '50 years of chemical engineering endeavour' and its foundation in 1908, the Kestner Evaporator & Engineering Co., Ltd., have published the *Kestner Golden Jubilee Book*, with a foreword by its founder and present Chairman, Mr. James Arthur Reavell. Mr. Reavell, who is a brother to the founder of the well-known compressor company in Ipswich, had met Paul Kestner, a Frenchman from Alsace, a few years earlier. Kestner had a number of inventions to his credit, including an evaporator, acid elevators and fans, and suggested that Reavell should take over the Empire rights (excluding Canada) for his inventions. He did, however, insist that any company set up by Reavell should include his name in its title. Reavell, with his background of mechanical engineering and his service in several firms manufacturing pumps, electrical and hydraulic machinery, and equipment for use in sugar refining, was familiar with the field of Kestner's inventions and accepted his proposal. To avoid confusion with his brother's firm, he was glad to use Kestner's name for the company he founded.

From its foundation, the Company has prospered and has expanded its activities into many fields of chemical engineering plant manufacture. It is difficult today, when the manufacture of chemicals in an acceptable form as a raw material for further manufacture and conveniently packaged for the general public is commonplace, to realise how ill-developed processing plant was 50 years ago. The processing of foodstuffs, such as the concentrating of fruit juices or other commodities where excessive heat during evaporation of the solvent or moisture content might damage the final product was in a primitive stage. But Kestner's climbing film evaporator solved this and other problems, and at the same time reduced fuel consumption.

The book is conveniently divided up into chapters dealing with the main branches of the Company's activities. They cover, in addition to evaporation, acid handling, the drying of solids and slurries, the use of kerbush and other acid resisting materials of construction, high temperature process heating, effluents and their treatment, together with descriptions of complete process plant installations and finally the manufacture of small pilot-scale plant for use in universities and schools.

Times of national emergency have the merit of sharpening inventive abilities and the most energising effect on those connected with the manufacture of badly needed commodities. This is true of the manufacture of chemical plant and through both world wars the Kestner Company was able to show how such problems could be solved as they presented themselves. The manufacture of amatol in the first world war was imperative to keep our batteries in action in France, and silica gel was essential to keep gun turrets habitable in the second world war. The large-scale production of both was made possible by special plant designed and manufactured by the Company. Thus through excellent design, a thorough knowledge of the principles involved and a readiness to use any new constructional material as it became available, such firms were, and still are, able to manufacture at an economic price, plant for the production of chemicals in large quantities in a high state of purity. The book, which is attractively produced, is well written and profusely illustrated.

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Natural gas developments

EXPANSION IN THE FRENCH GAS INDUSTRY—£8 MILL. RAISED

GAZ DE FRANCE has lately raised another 100 mill. francs (roughly £8 mill.) on the French Stock Exchange to finance further expansion of the gas industry in that country. The issue's main features are guaranteed capital and growing interest linked to the country's gas sales in the years to come. Why are investment and confidence in the French gas industry particularly justified at this juncture?

Over the past decade sales by Gaz de France have steadily risen by about 5% each year. New gasworks have been erected and old ones extended in Paris, Rouen and Strasbourg, but no fewer than 250 small units have discontinued production, bringing the total down from 460 to 243 units. Yet in ten years the output has risen from 8,800 mill. therms a year to some 11,000 mill. yearly of coke gas and oil refinery by-products.

Important link

Distribution of the gas by means of a grid between cities and villages has reached its most advanced stage in the Paris Basin, the North, East and Centre of France. The most important link is that between Lorraine and Paris which is 300 kilometres long and carries 1 mill. cu. metres daily at a pressure of 60 kg. per sq. cm., an achievement unsurpassed in Europe.

Storage of gas is now to be carried out in vast underground gasholders, the first of which is now installed at Beynes, near Versailles. At a depth of some 400 metres beneath the ground, roughly 150 mill. cu. metres may be stored at 45 kg. per sq. cm. pressure. This may be approximately described as equivalent to 700 conventional gasholders. Work is in progress to double the capacity of this installation within a short while.

These developments are spectacular, but the chief interest for the future among gas engineers in France resides in the natural gas originating in the South West region, notably at Lacq.

Natural gas is expected to be in regular domestic use by Parisians by 1962 when the pipe-line network is completed. By that date Gaz de France will be distributing 22,000 mill. therms of all-type gas—twice the present amount. Already the company has completed the first high pressure section of the natural gas pipe-line, that between Lussagnet and Angoulême and Nantes on the West Coast. Another section will be serving the Montluçon, Lyon and East Central regions by May 1959, while a branch of this section serving Dole will be ready by the end of the year.

Five-and-a-half years of drilling, research and discouraging results followed the original uncontrolled eruption of dangerously toxic gas in late 1950, having a high hydrogen sulphide content, and it was not until 1957 that the first purified Lacq gas entered the pipe-line.

Progress in the 18 months or so to January 1959 has been remarkable, and the daily output of Lacq is already well over 1 mill. cu. metres. By 1961 it is confidently expected that the daily output will have reached 13 mill. cu. metres of pure gas (20 mill. of unpurified). This figure of 13 mill. is three times the present national gas consumption.

Development beyond 1961 or 1962 must depend on the resources, and these are not calculated with any kind of accuracy. The factories at Lacq and St. Margaret estimate that there are at least 200,000 mill. cu. metres in the region, and feel that the accumulation may, in fact, be many times as much.

The exploitation of these reserves is undertaken entirely by one company, the Société Nationale des Petrolas d'Acquitaine. This firm's main problem now is not to overcome technical difficulties and setbacks, but to ensure a regular market for a perfectly finished product. They are no longer concerned with deterioration of the pipe-line, but with finding enough home and export customers.

Briefly, the plan is to divide the market in France into three: The 12 Departments of the South West grouped around Toulose and Bordeaux; the Paris Region; the rest of Metropolitan France. In the case of industrial consumers above a certain size, the marketing is the responsibility of Gaz du Sud Ouest around Toulouse-Bordeaux and Gaz France elsewhere.

For all the possibilities of the ne energy form (although in point of fa it was known to the Romans her abouts), in industry and electric production as well as in domestic u it is surprising to learn that natur gas is unlikely to be extended throug out the country, but only to tho towns and villages where a regul consumption is likely to reach a val that will make the pipe-line wor while. There must be reimburseme of financial outlay within reasonab time. Thus there will continue to b wide use of conventional gas tec niques in modernised form for a lo time to come.

In general, most provincial town and villages will have to increase the number of large consumers in orde to qualify for natural gas, and th long-term plans for de-centralisin industry will assist in this direction In this way gas from the Lacq regio will probably slowly replace coke ga throughout France. Even so natura gas is unlikely to be distributed to a town if adequate cheap coal or hydr power already affords abundance o another energy form; the economy o the nation as a whole will be take into account in every instance.

There exists a Ministerial Com mittee whose primary task is to study the geographical extension of the new energy form. This Committee has made a number of recommendation based on the estimated Lacq output figure of 13 mill. cu. metres daily, and in particular they propose that the South West should take 5.2 mill., East Central 3.4 mill., Paris Region 3.2 mill., West 1.2 mill.

Sahara gas

With the development of the new Franco-African community, the pre sence of natural gas as well as oil in the Sahara assumes new importance. Plans are already in hand to pipe oil from the Sahara via Marseilles and Gibraltar to France and the rest of Europe. Taking a long view it can not be doubted that Saharan natural gas will be similarly linked to the European Market whatever form of economic unity this Continent adopts.